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Effects of Food Processing on Residues of Ethylenebisdithiocarbamate Fungicides and Ethylenethiourea

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EFFECTS OF FOOD PROCESSING ON RESIDUES OF ETHYLENEBISDITHIOCARBAMATE FUNGICIDES AND ETHYLENETHIOUREA



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Tomatoes, carrots, and spinach were treated at application rates recommended for good agricultural practice and at 4 times these rates with two ethylenbis(dithiocarbamate) (EBDC) fungicides (Dithane M-45[R] and Dithane M-22[R]).

These crops were grown in keeping with good agricultural practice at two locations; East and West. The pesticide residues, the presence of a metabolite, ethylenethiourea (ETU) and the effects of standard food processing techniques on residues of both EBDC fungicides and ETU were determined.

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EFFECTS OF FOOD PROCESSING
ON RESIDUES OF TWO ETHYLENEDITHIOCARBAMATE (EDDC)
FUNGICIDES AND ETHYLENETHIOUREA (ETU)

by

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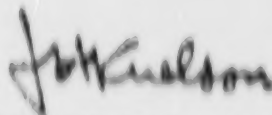
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FOREWORD

The many benefits of our modern, developing, industrial society are accompanied by certain hazards. Careful assessment of the relative risk of existing and new man-made environmental hazards is necessary for the establishment of sound regulatory policy. These regulations serve to enhance the quality of our environment in order to promote the public health and welfare and the productive capacity of our Nation's population.

The Health Effects Research Laboratory, Research Triangle Park, conducts a coordinated environmental health research program in toxicology, epidemiology, and clinical studies using human volunteer subjects. These studies address problems in air pollution, non-ionizing radiation, environmental carcinogenesis and the toxicology of pesticides as well as other chemical pollutants. The laboratory develops and revises air quality criteria documents on pollutants for which national ambient air quality standards exist or are proposed, provides the data for registration of new pesticides or proposed suspension of those already in use, conducts research on hazardous and toxic materials, and is preparing the health basis for non-ionizing radiation standards. Direct support to the regulatory function of the Agency is provided in the form of expert testimony and preparation of affidavits as well as expert advice to the Administrator to assure the adequacy of health care and surveillance of persons having suffered imminent and substantial endangerment of their health.

The significance of the occurrence of residues of pesticides or metabolites of pesticides in or on plants destined for human consumption must be considered with respect to its ultimate impact on human health. The present study evaluates the occurrence of residues of a widely used class of fungicide (ethylenebisdithiocarbamates) and the presence of a biologically active metabolite (ethylenethiourea) as a residue in food. The occurrence of the pesticide residue in several raw agricultural crops and the effects of commercial and home processing for consumption on the disappearance of the pesticide residue is evaluated with respect to several major food commodities upon which this pesticide class finds wide use.



John H. Knelson, M.D.
Director,
Health Effects Research Laboratory

ABSTRACT

Tomatoes, carrots, and spinach were treated at application rates recommended for good agricultural practice and at 4 times these rates with two ethylenebisdithiocarbamate (EBDC) fungicides (Dithane M-45[®] and Dithane M-22[®]). These crops were grown in keeping with good agricultural practice at two locations, East and West. The pesticide residues, the presence of a metabolite, ethylenethiourea (ETU) and the effects of standard food processing techniques on residues of both EBDC and ETU were determined.

Methods sensitive to 0.1 ppm EBDC and to 0.01 ppm ETU were used. All results were corrected for control values and for recovery of fortified samples analyzed concurrently.

As a direct result of the analytical procedures, EBDC was found to convert spontaneously to ETU during analysis. Average conversions of EBDC to ETU in control samples of the three crops fortified with Dithane M-45[®] ranged from 7-16%; conversions were 19.9 and 7.3% for tomatoes and spinach, respectively, when fortified with Dithane M-22[®].

On crops treated with Dithane M-45[®] and grown in the East, low levels of EBDC as a residue and of ETU were found in tomatoes and carrots; residues from spinach were appreciably higher. Residues in the Western crops, relative to those grown in the East, were higher on tomatoes and lower on carrots; EBDC residues were much higher on spinach. In no case were the tolerance levels of EBDC (Dithane M-45[®]) exceeded on carrots or tomatoes. With spinach, on which Dithane M-45[®] is not registered for

use, up to 62 ppm EBDC was found on the unwashed product. (Dithane M-45[®] was applied at the rate generally recommended for Dithane M-22[®] use on this crop). In the crops treated with Dithane M-22[®] (only at the Western location), average residues of EBDC on tomatoes did not exceed allowable tolerance levels. Again, residues on spinach were excessive in the unwashed crop.

Residues of ETU were noted on unwashed crops resulting from the use of both EBDC fungicides. The ETU residues were most notable on unwashed spinach from the Western location.

Washing in all cases removed significant amounts of both EBDC and ETU residues. However, residues of EBDC on western spinach exceeded tolerance levels even after washing. Washing also removed significant quantities of the ETU residue where it was noted on unwashed (Western) spinach. Further commercial food processing techniques decreased residues of EBDC significantly in all the crops and especially in spinach. However, ETU residues tended to increase with commercial food processing. Cooking of canned whole tomatoes for 5 and 60 minutes simulating home preparation followed this same pattern (reduced EBDC content - increased ETU content). While EBDC residues declined on normal commercial food processing, ETU residue levels increased. In concentrating tomato juice to tomato sauce and further to tomato paste (from crops treated with Dithane M-22[®]), ETU residues increased to an extent that appeared to be proportional to the degree of concentration or water loss. With western spinach treated with Dithane M-22[®], average ETU residues were appreciable

in all of the processed spinach. In cooking the spinach (again simulating household preparation of both frozen or canned product) ETU residues did not change appreciably.

The data, in part, further suggest that greater conversion of EBDC to ETU may occur in laboratory-fortified samples than in field-weathered samples.

This report was submitted by Technological Resources Inc., a subsidiary of Campbell Soup Company in fulfillment of Subcontract to Battelle Memorial Institute, Contract No. 68-02-1715 under the sponsorship of the U.S. Environmental Protection Agency. This report covers the period March 1, 1975 to July 31, 1976 and work was completed as of December 31, 1976.

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LIST OF ABBREVIATIONS

A	== acre
ai	== active ingredient
avg	== average
C	== Centigrade
CC	== Closed Cup
CI	== Chemical Impact
cm	== centimeter
EDC	== ethylenedithiocarbamate
EC	== emulsifiable concentrate
EI	== Electron Impact
ETU	== ethylenethiourea
F	== Fahrenheit
FP	== Flash point
g	== gram
gal	== gallon
GC or GLC	== gas chromatograph
GC/MS	== gas chromatograph/mass spectrographic analysis
ft	== foot
ha	== hectare
in	== inch
kg	== kilogram
lb	== pound
min	== minute
ml	== milliliter
mm	== millimeter
mv	== millivolt
ng	== nanogram
OC	== Open Cup
ppm	== parts per million
pt	== pint
ug or ug	== microgram
V	== volume
WP	== wettable powder

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SECTION I

INTRODUCTION

This study was undertaken to determine the effects of food processing on residues of two ethylenebisdithiocarbamate (ESDC) fungicides and a metabolite, ethylenethiourea (ETU), common to this class of fungicides. One of the fungicides was Dithane M-45[®] (zinc ion and manganese ethylenebisdithiocarbamate), a coordination product of manganese 16%, zinc 2%, ethylenebisdithiocarbamate 82% (also called mancozeb and Manzate 200[®]). The second fungicide was Dithane M-22[®], manganese ethylenebisdithiocarbamate (maneb, Manzate[®], Chem Neb[®]). [The major difference in the chemicals appears to be the presence of 2% zinc in Dithane M-45[®] and the absence of zinc in Dithane M-22[®].]

Three crops grown at Eastern and Western U.S. locations were treated with Dithane M-45[®]. Residues of the parent compound and metabolite were determined on the fresh and washed raw agricultural commodity, processed canned and frozen products. In addition, a cooking experiment was conducted to determine the stability of technical Dithane M-45[®] and the rate of formation and degradation of ethylenethiourea in boiling water.

Tomatoes, carrots and spinach were grown on the East Coast of the U.S. in Rancocas, New Jersey and on the West Coast at Davis, California. Experimental plots, which were arranged in a Latin Square design, were treated at two rates of application and included a control (untreated). Treatments, replicated three times, were made in accordance with the

current EPA Compendium of Registered Pesticide Uses (1976), where applicable, and at four times the registered rates. Tolerances of EBDC on tomatoes and carrots treated with Dithane M-45[®] are 4 and 2 ppm, respectively; Dithane M-45[®] is not registered for use on spinach. Tolerances of EBDC on tomatoes, carrots, and spinach treated with Dithane M-22[®] are 4, 7, and 10 ppm, respectively. Since Dithane M-45[®] is not registered for use on spinach, the 1 and 4-fold rates of 1.6 pounds and 6.4 pounds active ingredient per acre (1.79 and 7.17 kg/hectare) for Dithane M-22[®], which is registered for use on spinach, were used to apply Dithane M-45[®] to spinach. With this exception, crops were grown and processed in accordance with good agricultural and manufacturing practices.

Two of the three crops, tomatoes and spinach, grown at Davis, California were experimentally treated with Dithane M-22[®] to evaluate the effects of processing on residues of this fungicide. The Dithane M-22[®] study was conducted in essentially the same way as the Dithane M-45[®] study except that additional processing parameters on residues of EBDC and ETU were examined. The additional processing parameters included canning of whole tomatoes, simmering of tomatoes for two time intervals, preparation of tomato sauce and tomato paste, and cooking of frozen and canned spinach. Experiments were also conducted to determine effect of processing on conversion of residues of EBDC to ETU in whole tomatoes field-treated 4x the registered rate vs. those in a control sample laboratory-fortified with EBDC and ETU at the same level as in the field-treated sample.

SECTION II

MATERIALS AND METHODS

Each of the following treatments were replicated three times, and each replicate was analyzed in duplicate. All residue values are reported in ppm (parts per million). All results are corrected for control values and data on recovery of fortified samples.

GROWING AND TREATMENT OF CROPS

The tomatoes, carrots, and spinach were grown at two locations--Rancocas, New Jersey and Davis, California. After the plots were subdivided in a Latin Square design, they were treated with Dithane M-45[®] or Dithane M-22[®] at 0, 1, and 4 times the registered application rate (Note: Dithane M-45[®] is not recommended for use on spinach. The same dosage levels were used for both products). Table 1 presents details describing the growing, treatment, and harvesting of the three crops in the Eastern location, and Table 2 gives similar data for the 3 crops grown in the West. Samples from the Eastern Dithane M-22[®] applications were not analyzed.

PROCESSING OF CROPS

The following conditions were employed for processing the crops. Samples except for those canned, were frozen and stored at -20°F (-29°C) until analyzed.

Tomatoes

Washed and Unwashed Samples--

Fresh harvested California tomatoes, both unwashed and cold-water-washed, were frozen for shipment to the Camden, New Jersey laboratory

Table 1. Conditions for Growing, Treatment, and Harvesting of Crops Grown at Rancocas, New Jersey (Eastern location)

Treatment	Spinach ^a	Carrots	Tomatoes
1. Fertilizer	253 lb/A 10-10-10 disc'd before planting, 90 lb/A ammonium nitrate top-dressed on 5/23 and 6/10/75.	Fumigant methyl bromide 1 lb/100 sq. ft.	600 lb/A 10-10-10 disc'd in before planting.
2. Variety	Long standing Bloomsdale	Danvers 126	Campbell 17
3. Seeded	4/18/75	4/16 and 5/19/75	5/24/75
4. Herbicide	CIPC 0.5 lb ai/A and Veedex 2 lb ai/A applied preemergence 5/22/75.	none	Pebulate 4 lb ai/A before planting, Chloramben 4 lb ai/A granular broadcast.
5. Insecticides	Malathion 1 qt 57% EC/A, Diazinon 1/2 pt of 4 lb/gal EC/A. Combination applied with air blast sprayer 5/22 and 6/3/75.	Sevin 2 lb ai/A 7/11 and 7/26/75	Azinphosmethyl 0.25 lb ai/A on 5/10 5/26, 6/10 6/19/75. Carbaryl 1 lb ai/A plus endosulfan 0.5 lb ai/A 7/1, 7/11/75. Carbaryl 1 lb ai/A 7/26/75.
6. Fungicide	Dithane applied 6/9/75 1.6 lb ai/A (1.79 kg/ha) in 37 gal (140 liters) water/A. 6.4 lb ai/A (7.17 kg/ha) in 50 gal (189 liters) water/A.	Dithane 1.6 and 6.4 lb ai/A (1.79 and 7.17 kg/ha) 7/17, 7/24, 8/4, 8/13, 8/27, 9/10/75.	Dithane 1.6 and 6.4 lb ai/A (1.79 and 7.17 kg/ha) 7/8, 7/17, 7/24, 8/4, 9/16 applied in 50 gal (189 liters) water/A.
7. Harvested	6/19/75	9/30/75	9/22/75

^aCrop injured by smog during week of 5/19/75 (30 days prior to harvest).

Table 2. Conditions for Growing, Treatment, and Harvesting
of Crops Grown at Davis, California (Western Location)

Treatment	Spinach	Carrots	Tomatoes
1. Fertilizer	20-10-10 at 160 lb/A broadcast preplant	none	Nitrogen 20 lb (broadcast) preplant. Starter (9-24-0) 16 gal (67 liters) water/A. Two inches under seed.
2. Herbicide	Ro-nast (73.9%) Cycloctc 2/3 gal/A broadcast preplant	none	Trufmid 8.0 lb/A broadcast preplant
3. Insecticides	Spectracide (Diazinon 25%) 1/2 pt/A 1/13/76	none	Sevin (50% WP) 2.0 lb ai/A; Sulfur 40 lb/A 7/10, 7/31, 8/21/76.
4. Fungicides	Dithane applied 3/23/76, 1.6 lb ai/A (1.79 kg/ha) in 37 gal (140 liters) water/A and 6.4 lb ai/A (7.17 kg/ha) in 50 gal (189 liters) water/A.	Dithane 1.6 and 6.4 lb ai/A (1.79 and 7.17 kg/ha) 9/8, 9/16/76.	Dithane 1.6 and 6.4 lb ai/A (1.79 and 7.17 kg/ha) 8/22, 8/29, 9/5/76.
5. Harvested	4/2/76	9/22/76	9/10/76

for processing and analysis. Fresh-harvested New Jersey tomatoes were shipped direct to the laboratory. Washed tomatoes were prepared by cold-water washing for approximately three to five minutes with continuous flow of cold tap water with agitation. Washed and unwashed samples were macerated with dry ice to maintain a free-flowing powder and held at -20°F (-29°C) until analyzed for EBDC and ETU.

Canned Juice and Tailings--

In the pilot plant, approximately 35 pounds (15.9 kg) from each treatment were cold-water washed twice, trimmed where necessary, and then macerated in a Hobart mixer. The macerate was heated with stirring to 180°F (82°C) in a steam-jacketed kettle. Heated tomatoes were passed through a Langsenkamp extractor fitted with an 0.027 screen. Resultant purees were heated to 200°F (93°C), canned, processed for 20 minutes in boiling water, cooled, and cased. Tailings from each extraction (seeds and skin) were collected and frozen.

Sauce--

Tomato juice was evaporated at 60-70°C under 25-psf vacuum to yield a product with about 15% solids.

Paste--

As in sauce above, but product was concentrated to 30-35% solids.

Canned Whole Tomatoes--

Washed tomatoes were immersed in hot water (ca. 70°C) for five minutes. After the skins were removed, the tomatoes were transferred to cans and processed at 240°F (116°C) for 45 minutes.

Carrots

Unwashed--

Fresh-harvested carrots were vigorously washed with cold water to remove dirt.

Washed--

Skins from the carrots above were removed by hand with a wire brush and cold-water washed.

Diced--

Carrots were passed through a washer and abrasive peeler and diced (1/4") in Pilot Plant.

Frozen--

Diced carrots from above were blanched for four minutes in 200°F (93°C) water, drained and frozen.

Canned--

Diced carrots and hot 190°F (88°C) water were added to No. 1 cans, sealed, and processed in a retort for 40 minutes at 240°F (116°C).

Spinach

Unwashed (rinsed)--

Unwashed, fresh-harvested spinach was cold-water rinsed to remove dirt.

Washed--

Fresh spinach was washed with cold water.

Frozen--

Washed spinach from above was blanched for three minutes at 190°F (88°C) in a steam-jacketed kettle, chopped, and frozen.

Canned--

Washed spinach from above was blanched for three minutes at 190°F (88°C) in a steam-jacketed kettle, chopped, canned, and processed for 55 minutes in a pressure cooker at 240°F (116°C).

Additional Processing

To determine the effect of cooking, canned whole tomatoes were cooked for 5 minutes, and the resultant solids and drained water were each analyzed for EBDC and ETU.

METHODS OF ANALYSIS

EBDC

The analytical method employed for determining residues of EBDC was described by Keppel (1971). For the crops studied, the sensitivity is 0.1 - 0.2 ppm of EBDC with an average recovery of 86.6%. Standards for calibration and recovery studies were prepared as described by Gordon, et al. (1967).

ETU

The analytical method used for determining ETU, the Haines-Adler Method (1973) (Rohm and Haas Test Method 1881-1), as modified by Technological Resources Inc., is presented in detail in Appendix A. It is essential that all sources of contact with rubber be eliminated. It was found that the addition of a small amount of trichloroacetic acid to the extraction solvent improved the recovery of ETU from spinach and carrot matrices. The GC column packing and conditions for separation of ETU were also modified. The major advantages of the modified packing are more stable baseline, better separation of ETU from possible crop interferences, and improved GLC response to the S-butyl derivative. There

should be no delay in processing samples from time of extraction to the formation of the S-butyl derivative. Minimum time of exposure to DMF is also critical. Samples may be held overnight for gas chromatography after being dried. The sensitivity of the modified method is 0.01 ppm for carrots, spinach, and tomatoes.

A number of samples of tomatoes and spinach were sent to Dr. Ralph Nash of USDA, Beltsville, Maryland to determine the ETU residues by an independent method. Unfortunately, Dr. Nash separated and analyzed the liquid and solid portions of the crops while the method used for this report analyzed the entire sample. The results, however, while not fully comparable, are quite similar:

Commodity	Rep. No.	ETU found (Nash)	ETU found (TRI)
Western spinach unwashed	11	0.26	0.32
Western spinach washed	11	0.07	0.01
Eastern spinach canned	11	0.16	0.18
Western spinach canned	11	0.77	0.60
Western tomato canned	1	0.07	0.16
Eastern tomato canned	11	0.05	0.05

TYPICAL ANALYSIS OF DITHANE (M-45 or M-22^(B)) for EBDC and ETU

The formulated Dithane M-45^(B) was analyzed for EBDC (ai) and ETU content. The results¹ are shown below:

Product	Sample No.	% ai	% ETU
Dithane M-45 ^(B)	2-0530-1	86.9	0.05
Dithane M-45 ^(B)	2-0530-2	83.8	0.11
Dithane M-22 ^(B)	2-4502-1	86.9	0.02
Dithane M-22 ^(B)	2-4502-2	89.7	0.01

¹Analysis of the same samples by Rohm and Haas gave 85.7% EBDC and 0.02% ETU for sample 2-0530-1, and 83.3% EBDC and 0.05% ETU for sample 2-0530-2.

CALCULATION OF DATA

The active ingredient (EBDC) was determined by CS_2 generation and expressed as Dithane M-45 through use of the following calculation (assuming the M.W. of Dithane M-22 to be 265.38):

$$\text{wt. } \text{CS}_2 \times 1.78 = \text{wt. Dithane M-45}^{(R)}$$

The residues of parent compound in this study were calculated as EBDC (M.W. = 210):

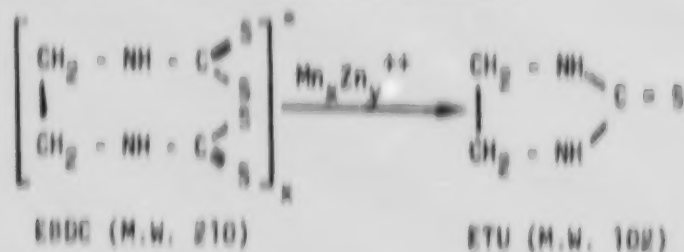
$$\text{wt. } \text{CS}_2 \times 1.38 \text{ and } 1.74 = \text{wt. EBDC}$$

The factors 1.78 and 1.38 were determined as follows:

$$\frac{\text{M.W. Dithane M-45}}{2 \times \text{M.W. } \text{CS}_2} = \frac{\text{M.W. Maneb} + 2\% \text{ Zn}}{2 \times \text{M.W. } \text{CS}_2} = \frac{265.38 + 5.31}{152.26} = 1.78$$

$$\frac{\text{M.W. EBDC}}{2 \times \text{M.W. } \text{CS}_2} = \frac{210}{152.26} = 1.38$$

In a similar calculation for active ingredient of Dithane M-22/ CS_2 (factor $\frac{265.38}{152.26} = 1.74$) was made. As shown in the following equations, the theoretical yield of ETU from EBDC is 48.6%:



$$\frac{\text{M.W. ETU}}{\text{M.W. EBDC}} = \frac{102}{210} = 0.486 \text{ or } 48.6\%$$

METHOD OF FORTIFICATION OF CROPS

The following procedure was typically followed: Ten mg Dithane M-45^(R) (or Dithane M-22^(R)) was added to 100g starch and the mixture thoroughly

dispersed by stirring and shaking. The starch mixture was analyzed for percent active ingredient, and 60 ug EBDC/g starch was found. Based on this concentration of EBDC, the appropriate weight of starch for fortification was added to the sample. This standard method of fortification is essentially the one described by Gordon et al. (1967), except that starch was substituted for Attapulgate clay.

For fortification with ETU, the chemical was made up in water, and the appropriate volume of the solution was added to the crop. A detailed description of the preparation and procedures is given as part of the ETU analytical procedure (Appendix A).

SECTION III

RESULTS AND DISCUSSION

The application of the registered level of the fungicides (1x treatment) was designed to simulate good agricultural practice. Fungicide treatments at 4 times the registered level (4x treatment) were designed to expose effects that might be missed with the normal level of application. In both cases (and in the untreated controls or checks) the processing (excluding the home cooking experiment) simulated good commercial practice. Results obtained from this study, which include analyses at each processing point of the three crops from the time of harvest, should therefore provide a realistic insight into the presence of EBOC and ETU at the various processing stages.

Tables containing the analytical data have been assigned Roman numerals, and these tables have been placed immediately following the text or in the Appendices. A listing of the tables is presented ahead of the Introduction (see Contents). Summary tables, which give data on sub-studies, generally give average residue values. Individual analytical values, which are given in tables in the Appendices, have the same Roman numerals as the Summary Tables and are followed by additional figures. For example, the individual data from the Summary Table I may be found in Tables having i as the first letter of the Table Number.

RESIDUES FROM DITHANE M-45

Eastern Crops

As shown in Table 3, low levels of the parent compound (EBDC) and the metabolite, ETU, were found on carrots and tomatoes grown in the East. After processing both crops at the 1x rate of treatment, virtually none of the parent compound (0.1 ppm or less) remained. During heat processing of the 1x-treated tomatoes and carrots, the concentration of ETU increased from <0.01 ppm to an average of 0.03 ppm in canned tomato juice and canned carrots; the ETU concentration did not increase in the frozen samples of carrots and tomatoes. The 4x treatment of unwashed tomatoes and carrots contained parent compound at a concentration of 0.8 ppm. The average ETU content of canned carrots and juice of tomatoes grown in the East was 0.05 and 0.10 ppm, respectively, for the 4x rates. Spinach grown in the East contained appreciably higher levels of EBDC and ETU than those in carrots and tomatoes in both the 1x and 4x treatments.

Western Crops and Comparison with Eastern Crops

Table 3 also presents the residue data from crops grown in the West. Residues of EBDC in West-grown tomatoes (up to 7.1 ppm at the 4x rate) were greater than those on tomatoes grown in the East; on West-grown spinach, EBDC residues (up to 219.3 ppm at 4x rate) were great, although they were virtually absent in canned spinach and low (0.6 to 2.0 ppm at 4x rate) in frozen spinach. Residues of ETU in West-grown tomatoes were about the same as those in the Eastern tomatoes except that amounts in the canned tomatoes were somewhat greater (up to 0.13 ppm at 4x rate); ETU residues in carrots were generally less than 0.01 ppm

(less than in Eastern carrots); ETU residues in Western spinach (up to 3.74 ppm) were far greater than corresponding residues in the Eastern spinach. Thus, the study shows that very low residues may be expected on carrots, low residues on tomatoes, and relatively high residues on spinach when Dithane M-45[®] is applied at the 1x rate.

Except for unwashed Western-grown tomatoes from the 4x rate, residues of EBDC on tomatoes and carrots (East or West) from the 1x and 4x treatment with Dithane M-45[®] were far below their respective tolerances of 4 and 2 ppm.

Effects of Washing and Canning

Analyses of EBDC and ETU of unwashed, washed, and processed canned products are shown in Table III (Appendix D), along with percent of ETU converted from EBDC during this processing. The value for percent conversion is based upon the amount of ETU found in the sample relative to the theoretical quantity of ETU which could be obtained from the residue of EBDC present prior to processing. These data indicate that washing removes a significant amount of residue of EBDC (up to 87%). The data also indicate that some of the EBDC residue is converted to ETU during heat processing with the percent conversion being higher for East-grown than West-grown crops. Average conversions ranged from 2.6 - 70.4% with an overall average of 26.6%. However, levels of EBDC residues on tomatoes and carrots appear to be too low to assign any real significance to the conversion values obtained.

Residues in tomato tallings (East or West) at the normal application rate were very low (Tables IA-1-C and IB-1-C in Appendix B); at the

4-fold rate, respective residues of EBDC and ETU were 0.5 and 0.01 ppm in the East and 1.4 and 0.04 in the West.

Conversion of EBDC (Added as Dithane M-45[®]) to ETU Owing to Analytical Method and Matrices

In order to determine conversion of EBDC to ETU due to the analytical method and different matrices, control samples fortified with EBDC and ETU were analyzed for ETU and percent conversion calculated. The results are shown in Tables IV (Appendix D). The individual conversions ranged from 1.6% to 46.6%. These results indicate that on the average there is 7-16% conversion of added EBDC to ETU in the ETU analysis of the tomatoes, carrots, and spinach grown in the East and West.

In these fortification experiments, 0.06 ppm ETU was added to all samples to improve the detectability of low levels of ETU by the flame photometric detector. Response of the detector is proportional to the square of the concentration of sulfur-containing compounds, so response increases appreciably as the ETU concentration is increased.

Conversion of EBDC to ETU due to the analytical method was determined in tomato juice when fresh EBDC alone was added to a control sample. The conversion of EBDC to ETU was 16.6%. This conversion is comparable to the conversion found in control samples of tomato juice when both EBDC and ETU are added to the control sample matrix.

ETU Formation from Weathered Residues and from Added Dithane M-45[®]

Analyses of treated crops indicate that there is much less conversion of EBDC to ETU from weathered residues of technical Dithane M-45[®] than from Dithane M-45 that is laboratory-fortified. The maximum theoretical

concentration of ETU from EBDC found on washed crops was calculated ($\text{EBDC} \times 0.46 = \text{Max. ETU}$), and the ETU actually found was expressed as a percentage of the 100% theoretical conversion. For Eastern- and Western-grown tomatoes and carrots and for Eastern-grown spinach, the ETU residues from weathered samples did not exceed 0.01 ppm, so significant values for percent conversion could not be obtained for these crops. However, for Western-grown spinach, residues of EBDC and ETU were large enough to calculate percent conversions. These data on weathered residues and similar data from Dithane M-22[®] applications (to be discussed later) follow:

Crop	Loc.	Treat.	Amount Found EBDC	Theor. Max. Amt. ETU Obtainable	Amount Found ETU*	% Conversion
Washed Spinach	West	1x(M46)	9.7	4.71	0.02	0.4
Washed Spinach	West	4x(M-46)	58.7	28.53	0.08	0.3
Washed Spinach	West	1x(M-22)	35.2	16.9	0.06	0.4
Washed Spinach	West	4x(M-22)	192.4	92.4	0.41	0.4

*For sake of discussion, the values under "Amount Found ETU" are assumed to arise from conversion of EBDC to ETU.

The above percent conversions of EBDC to ETU for weathered residues on West Coast spinach (0.38% average) are far below the 7.3% conversion of EBDC (added as Dithane M-46[®]) to ETU for the same crop (See Table IV).

An experiment was conducted to determine the rate of formation (and breakdown) of ETU from technical EBDC in hot water. Distilled water was buffered to pH 6.21 with citric acid/disodium phosphate. This pH is

representative of the mid-range of acid foods. A portion of the buffer solution served as a control. The two solutions were brought to a boil in two-quart stainless-steel pots, and Dithane M-45[®] was added to one of them (initial concentration EBDC 6.2 ppm). Aliquots were withdrawn from the boiling Dithane M-45[®] water solution after ca. 0.5, 30, 60, and 90 minutes. Similar aliquots were withdrawn from the boiling control solution at 0 and 60 minutes. The aliquots of water were placed in 4-oz (113-ml) Nalgene bottles and rapidly frozen in a dry ice-acetone bath. Time involved for complete freezing of the samples was about 10 minutes. Samples were thawed for analyses of EBDC and ETU with a warm water bath.

The degradation of EBDC in boiling water and the formation of ETU occurred very rapidly (Table VI in Appendix D). On continued boiling for 60 minutes, about 56% of the ETU degraded. The apparent increased conversion at 90 minutes is attributed to concentration factors of boiling and withdrawal of aliquots.

RESIDUES FROM DITHANE M-22[®] (maneb)

Tomatoes, Residues at the Various Processing Stages

Table 4 summarizes the average residue data obtained from tomatoes grown in Davis, California and treated with Dithane M-22[®] at the 1x (registered) and 4x levels. Untreated (check) samples were included as controls. For the washed and unwashed tomatoes, levels of residues were similar to those reported for Dithane M-45[®] West Coast tomatoes except for the higher ETU residue (0.10 ppm) in the 4x treatment of the washed tomatoes.

As with Dithane M-45, washing reduced the residues of EBDC. Canned whole tomatoes had some residues (1x treatment EBDC 0.3 ppm, ETU 0.02; 4x treatment EBDC 0.4, ETU 0.07). When this commodity was cooked for 5 and 60 minute intervals, the EBDC residue decreased to 0.1-0.3 ppm, and the ETU residue increased to 0.06-0.07 ppm for the 1x treatment and was 0.06-0.11 for the 4x treatment.

Residues in canned tomato juice (ca. 6% solids) were similar to those reported for the West Coast product in the Dithane M-45 study and are slightly larger than those that were found in canned whole tomatoes. For the 1x treatment, residues were EBDC 0.4 ppm, ETU 0.04; 4x treatment EBDC 0.8, ETU 0.12.

The tomato sauce, which based on its solids content of about 15% may be considered an approximately 2½-fold concentrate of tomato juice, contained in the 1x treatment 0.8 ppm EBDC and 0.11 ppm ETU and in the 4x treatment 2.7 ppm EBDC and 0.22 ppm ETU. The increases in residues

over those in the tomato juice samples appear to be mainly a concentration effect; i.e., residues are about 2½ times those in the juice.

The tomato paste (ca. 30-35% solids) may be considered a further 2-fold concentrate of the tomato sauce. Again, the increase in residues appears to be mainly a concentration effect. Residues in the 1x treatment were EBDC 2.2 ppm and ETU 0.37; in the 4x treatment, EBDC 3.7 and ETU 0.46. These values are about double those found in the sauce.

The increase in ETU content, which appears to accompany the high temperature (ca. 116°C) used in canning, probably occurs to a much lesser extent in the lower-temperature concentration (ca. 70°C vacuum) used to prepare tomato sauce and tomato paste.

Spinach, Residues at the Various Processing Stages

Table 4 summarizes the average residue data obtained from spinach grown in Davis, California and treated with Dithane M-22[®] at the 0, 1x, and 4x levels. As with Dithane M-45[®]-treated West Coast spinach, residue levels were high (1x treatment EBDC 35.2-86.6 ppm, ETU 0.05-0.24; 4x treatment EBDC 186.8-192.4, ETU 0.05-0.24). In most cases, washing reduced the residue levels.

Blanching and freezing greatly reduced the EBDC residues on the washed spinach (1 and 4x treatments 0.4 and 0.9 ppm, respectively) but increased the ETU residues (1 and 4x treatment 2.00 and 0.89, respectively). After cooking and simmering the blanched frozen spinach for 5 minutes, residues of EBDC were lowered (0.2-0.4 ppm); ETU residues remained about the same (1 and 4x treatments 0.76 and 1.44, respectively). The water

drained from the blanched frozen cooked spinach had no EBDC residue; ETU residues were 0.65 and 0.58 ppm for the 1x and 4x treatments, respectively.

Canned spinach contained <0.1 ppm EBDC residues; ETU residues were 1.82 and 2.50 ppm for the 1 and 4x treatments. After cooking and simmering the canned spinach for 5 minutes, ETU levels were 2.12 and 2.26 ppm for the 1 and 4x treatments; ETU residues in the drained water from this cooking were 1.03 and 0.58 ppm for the 1 and 4x treatments, respectively.

The pattern of increased levels of ETU and very low levels of EBDC in canned products has continued with the spinach samples.

Residues from Whole Washed Tomatoes Fortified with EBDC and ETU

Control (untreated) whole washed tomatoes were fortified with amounts of EBDC and ETU found in whole tomatoes from the 4x field treatment with Dithane M-22[®] to compare residues from laboratory-fortified samples with those from the 4x field treatment. Fortifications were made with 0.5 ppm EBDC alone, with 0.07 ppm ETU alone, and with 0.5 ppm EBDC and 0.07 ppm ETU together. The data are summarized in Table VII (Appendix D) along with the residue data from whole tomatoes from the 4x treatment in the field.

In general, the residue data from the samples fortified with both EBDC and ETU agree reasonably well with the data from the field-treated samples.

The sums of the EBDC and ETU residues from the individual fortifications with EBDC and ETU also appear to be reasonably similar in magnitude to the EBDC and ETU residues found in the samples fortified with both chemicals.

Conversion of Added EBDC to ETU in Tomatoes and Spinach

The conversion of EBDC to ETU owing to analytical method and different matrices was determined as with Dithane M-45^(D) (see Table IV in Appendix D) by fortifying control samples with EBDC and ETU and analyzing for EBDC and ETU to determine percent conversion. A summary of the data is given in Table V.

Percent conversions of EBDC to ETU ranged from 1.7 to 50% for the washed and unwashed tomatoes and canned whole tomatoes and tomato juice, with an average conversion of 19.9%. This result agrees reasonably well with the 16% average conversion for West Coast tomatoes found in the Dithane M-45^(D) study.

For the unwashed, washed, frozen, and canned spinach, the percent conversion of EBDC to ETU ranged from 0.0 to 9.3% and averaged 5.4%. Again, there is reasonably good agreement of this value with the 7.3% average conversion for West Coast spinach found in the Dithane M-45^(D) study.

Confirmation of ETU by GC/MS

In addition to the quantitative check performed by Dr. Nash, ETU results on canned spinach control Rep III, 1x Rep I and 4x Rep III were confirmed by GC/MS. These analyses were performed on different subsamples than shown in Table 1C-4-D. The Hewlett-Packard Model 5980A mass spectrometer with an all-glass GC interface was employed.

The spectra for a 50 n sample of 5-n-Butyl ETU in the EI mode produced major clusters of ions around m/e 102 and 129. In the CI mode with methane, major ions at 159 (P + 1) and 187 (P + 29) were produced. These spectra along with those obtained for samples noted above are

shown in Appendix E, Tables I thru VIII. The samples of treated spinach contained a compound whose spectra and retention time matched the standard.

The same samples were also analyzed for ETU by the GLC method described in Appendix A. The table below compares the quantitative GC and GC/MS analyses performed.

Sample	ppm ETU			
	FPD (FFAP)	CI (OV-101)	CI (FFAP)	EI (FFAP)
Canned Spinach, Control, Rep III	<0.01	0.01	<0.01	<0.01
Canned Spinach 1x M-45, Rep I	1.03	0.83	1.04	1.02
Canned Spinach 4x M-45, Rep III	3.4	4.40		4.17

The MS quantitation was based ratios of single ion monitoring of sample vs. standard ions at mass 102 and 129 for EI and mass 159 and 187 in CI mode.

An early unknown eluter was detected in the 4x spinach sample by GC/MS in EI mode. The major ions of the unknown were at mass 116. No additional work toward identification of this compound was conducted. However, we intend to complete the identification, if possible, in the near future.

RESIDUE DATA FROM FIELD TREATED CROPS

These data are presented in Tables 3 and 4.

A summary of the recovery data is given in Table VIII (Appendix D).

Table 3. SUMMARY OF DATA ON RESIDUES OF EBDC AND ETU AT VARIOUS PROCESSING STAGES FROM TOMATOES, CARROTS, AND SPINACH GROWN AT EAST AND WEST COAST LOCATIONS AND TREATED WITH DITHANE M-45¹ AT 0, 1, AND 4 TIMES THE REGISTERED RATES. EACH RESULT IS AVERAGE OF AT LEAST SIX DETERMINATIONS.

Commodity	Process Stage	Application Rate	East Coast		West Coast	
			EBDC	ETU	EBDC	ETU
A. <u>Tomatoes</u>	Unwash	Check	<0.1	<0.01	<0.1	<0.01
	Unwash	1x	0.3	<0.01	2.1	0.01
	Unwash	4x	0.8	0.02	7.1	0.02
	Wash	Check	0.1	0.01	<0.1	<0.01
	Wash	1x	0.2	<0.01	0.6	0.01
	Wash	4x	0.3	0.01	0.9	0.01
	Tailings	Check	0.1	<0.01	-	<0.01
	Tailings	1x	0.2	<0.01	0.4	<0.01
	Tailings	4x	0.6	0.01	1.4	0.04
	Juice Canned	Check	0.1	0.01	<0.1	0.01
	Juice Canned	1x	<0.1	0.03	0.6	0.11
	Juice Canned	4x	0.1	0.10	1.8	0.13
B. <u>Carrots</u>	Unwash	Check	<0.1	<0.01	<0.1	<0.01
	Unwash	1x	0.6	<0.01	0.1	0.01
	Unwash	4x	0.8	0.01	0.4	<0.01
	Wash	Check	<0.1	<0.01	<0.1	<0.01
	Wash	1x	0.3	<0.01	0.1	0.01
	Wash	4x	0.7	<0.01	0.4	<0.01
	Diced	Check	<0.1	<0.01	<0.1	<0.01
	Diced	1x	0.1	<0.01	0.1	<0.01
	Diced	4x	0.4	<0.01	0.1	<0.01
	Frozen	Check	<0.1	<0.01	<0.1	<0.01
	Frozen	1x	<0.1	<0.01	<0.1	<0.01
	Frozen	4x	<0.1	0.01	<0.1	<0.01
	Canned	Check	<0.1	<0.01	<0.1	<0.01
	Canned	1x	<0.1	0.03	0.1	<0.01
	Canned	4x	<0.1	0.06	0.1	<0.01
C. <u>Spinach</u> ¹	Unwash	Check	0.2	<0.01	0.3	0.01
	Unwash	1x	2.4	<0.01	61.9	0.34
	Unwash	4x	6.4	0.01	219.3	1.41
	Wash	Check	0.1	<0.01	0.2	<0.01
	Wash	1x	1.6	<0.01	9.7	0.02
	Wash	4x	2.7	0.01	59.7	0.08
	Frozen	Check	<0.1	<0.01	0.1	0.01
	Frozen	1x	0.1	0.04	0.6	0.60
	Frozen	4x	0.4	0.07	2.0	3.74
	Canned	Check	0.1	0.01	0.1	0.01
	Canned	1x	<0.1	0.18	0.1	0.71
	Canned	4x	<0.1	0.44	0.1	2.66

¹ Dithane M-45[®] is not registered for use on spinach. Rates of 1.6 and 6.4 lb ai/acre were applied to match those of Dithane M-22[®], which is registered for use on spinach.

Table 4. SUMMARY OF DATA ON RESIDUES OF EBDC AND ETU AT VARIOUS PROCESSING STAGES FROM TOMATOES AND SPINACH GROWN IN THE WESTERN U.S. AND TREATED WITH DITHANE H-22 AT 0, 1, AND 4 TIMES THE REGISTERED RATES. EACH RESULT IS AVERAGE OF AT LEAST SIX DETERMINATIONS

Commodity	Process Stage	Application Rate	EBDC	ETU
A. <u>Tomatoes</u>	Unwashed	Check	0.2	0.01
	Unwashed	1x	2.4	<0.01
	Unwashed	4x	8.0	0.02
	Washed	Check	0.1	0.02
	Washed	1x	0.3	<0.01
	Washed	4x	0.6	0.09
	Canned Whole	Check	<0.1	0.01
	Canned Whole	1x	0.3	0.02
	Canned Whole	4x	0.4	0.07
	Juice Canned	Check	<0.1	0.02
	Juice canned	1x	0.4	0.04
	Juice Canned	4x	0.8	0.12
	Canned Whole Tomatoes, cooked and simmered for 5 minutes	Check	<0.01	<0.01
		1x	0.2	0.06
		4x	0.3	0.06
	Canned Whole Tomatoes, cooked and simmered for 60 minutes	Check	<0.1	<0.01
		1x	0.1	0.07
		4x	0.3	0.11
	Sauce	Check	<0.1	0.06
	Sauce	1x	0.8	0.11
	Sauce	4x	2.3	0.22
	Paste	Check	0.1	0.04
	Paste	1x	2.2	0.37
	Paste	4x	3.7	0.46
B. <u>Spinach</u>	Unwashed	Check	0.2	<0.01
	Unwashed	1x	86.6	0.24
	Unwashed	4x	186.6	0.73
	Washed	Check	0.1	<0.01
	Washed	1x	36.2	0.06
	Washed	4x	192.4	0.41
	Blanched Frozen	Check	<0.1	0.01
	Blanched Frozen	1x	0.4	0.76
	Blanched Frozen	4x	0.9	1.44
	Blanched Frozen Spinach, cooked and simmered for 5 minutes	Check	<0.1	<0.01
		1x	0.2	0.76
		4x	0.4	1.44
	Drained Water from Blanched Frozen Cooked Spinach	Check	<0.1	<0.01
		1x	<0.1	0.66
		4x	<0.1	0.68
	Canned	Check	<0.1	0.01
	Canned	1x	<0.1	1.82
	Canned	4x	<0.1	2.50
	Canned Spinach, cooked and simmered for 5 minutes	Check	<0.1	0.01
		1x	<0.1	2.12
		4x	<0.1	2.26
	Drained Water from Canned Spinach, cooked and simmered for 5 minutes	Check	<0.1	<0.01
		1x	<0.1	1.03
		4x	<0.1	0.58

SECTION IV

REFERENCES

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APPENDIX A

Detailed Analytical Procedure Used to Determine ETU¹

¹Modified Rohm & Haas Co. Procedure - Test Method 1881-1, July 29, 1978

This test method is a comprehensive procedure for the determination of trace levels (to about 0.01 ppm*, minimum) of ethylenethiourea (ETU).

Ethylenethiourea (ETU, 2-imidazolidinethione) is a suspected carcinogen and is a possible decomposition product of certain ethylene bisdithiocarbamate fungicides, among them Dithane. This test method was first developed to determine whether potatoes, including those treated with Dithane, were free from ETU residue. It has since been extended to include all Dithane-treated food crops. The method has also been successfully used to determine ETU residue in urine.

Method

ETU is extracted from finely divided samples with methanol and acetone; the resulting slurry is filtered and concentrated, then partially purified by passing it through an alumina column. A chromatographable S-butyl ETU derivative is prepared by mixing the column eluate with dimethyl formamide (DMF), evaporating the more volatile solvents, and then adding 1-bromobutane, sodium borohydride, and sodium hydroxide. A portion of this solution is acidified, evaporated to dryness, and redissolved in sodium hydroxide solution. The derivative is extracted from the basic solution with benzene, and the benzene extract is separated gas chromatographically, using a 6-foot, glass column packed with [5% KOH and 10% Carbowax 20M liquid phase on 80/100 Gas Chrom Q support.]* The ETU derivative is detected by a sulfur-sensitive, flame-photometric detector.

The ETU content is determined by comparing peak heights of samples and control samples to standard solutions of known S-butyl ETU content.

*As modified by MDG/WFP

2

A recovery factor is used to correct the ETU content of samples and is determined by adding known levels of ETU to portions of the untreated control samples, and carrying these "spiked" controls through the entire analysis.

Safety

Avoid contact with all toxic or corrosive liquids. In case of skin contact, wash the affected area immediately with copious quantities of water. In case of eye contact, similarly wash, and obtain medical assistance.

Ethylmethiourea (ETU) is suspected of causing cancer. Scrupulously avoid breathing ETU dust, or allowing skin contact with it or its solutions; use rubber gloves when handling. In case of contact, immediately wash the affected area with copious quantities of water, followed by soap and water. After handling ETU or its solutions, and especially before eating, drinking or smoking, wash the hands thoroughly with soap and water. Remove and wash any contaminated clothing before using again.

S-Butyl Derivative of ETU--The toxicity of this material is unknown, therefore, handle this material as though it were toxic; the same as ETU.

Acetone--Flammable (FP 0°F, CC). Moderately toxic by inhalation and causes skin irritation.

Benzene--Flammable (FP 12°F, CC). Highly toxic by ingestion, inhalation, and skin absorption. Tolerance, 25 ppm in air.

1-Bromobutane--Flammable (FP 75°F, CC). Probably toxic. Handle this material in a hood; mild lachrymator.

Dimethylformamide (DMF)--Moderately flammable (FP 136°F, CC). Highly toxic by skin absorption and ingestion; irritant to skin, eyes and mucous membranes.

Methanol--Flammable (FP 54°F, CC). Toxic by ingestion, skin absorption and inhalation.

Hydrochloric acid--Corrosive liquid, highly toxic by ingestion and inhalation; strong irritant to eyes and skin.

Sodium Borohydride--Reacts with water and solutions of acid or metallic salts to produce hydrogen (a flammable gas) and sodium hydroxide (a corrosive liquid). Dangerous fire risk. Store this material in a desiccator. Toxic through ingestion and skin contact; avoid breathing its dust.

Sodium hydroxide--This material and its solutions are corrosive. Highly toxic by ingestion and inhalation; strong irritant to skin, eyes and mucous membranes.

Petroleum Ether--Flammable (FP -40°F). Highly toxic by ingestion, inhalation, and skin absorption.

Hexane--Flammable (FP -9°F). Moderately toxic by inhalation and ingestion.

Apparatus

Aluminum Dishes, throw-away--5 cm diameter x 1.5 cm depth.

Balance--Top-loading, 1200 g capacity. Mettler Model P-1200 or equivalent.

Balance--Analytical.

Beakers--50 ml, 100 ml, 250 ml.

Bottles, Amber Glass--with aluminum-foil-lined screw caps. Various sizes.

Bottles, Plastic--Squeeze-type with capillary tip, 500 ml & 1000 ml.

Buchner Funnels, Coors size 4 (126 mm), Porcelain.

Burette, 50 ml with Teflon stopcock--Certified, NBS.

Centrifuge--Capable of 2000 rpm, with head capable of accepting Mini Vials.

Desiccator--with moisture indicating desiccant.

Dropping Pipettes, Capillary, Disposable--2 and 4 ml. Kimble Cat. No. 72020 and 72060, or equivalent.

Erlenmeyer Flasks--250 ml.

Erlenmeyer Flasks, with a $\frac{1}{2}$ 24/40 joint. 500 ml, 1000 ml.

Erlenmeyer Flasks, Bantamware--with $\frac{1}{2}$ 14/20 joint. 50 ml.

Filter Flasks--250 ml, 500 ml, 1000 ml.

Filter Funnel--Fine porosity, 30 ml.

Filter Paper--Whatman No. 1 & No. 541 or equivalent. To fit Buchner funnel (11 cm).

Freezer--Set to -5°C or colder.

Gas Chromatographic Columns--6 ft x $\frac{1}{4}$ in, glass, packed with (10% Carbowax 20M + 5% KOH)* liquid phase on Gas Chrom Q support.

Gas Chromatographic Instrument--(Micro-Tek 220,)* with Tracor flame photometric detector and 0-1 mV recorder.

Graduated Cylinders--25 ml, 50 ml, 100 ml.

Graduated Cylinders--with ground-glass stoppers, 10 ml.

*As modified by MDG/WFP

High-Speed Blender--Waring Blender or equivalent, with 1-pint glass container, screw-type metal lids. Replace all rubber gaskets with Teflon.

Liquid Chromatographic Columns--2.2 cm diameter x 10 cm length
2.2 cm diameter x 40 cm length
1.0 cm diameter x 26 cm length,
with 200 ml reservoir and a 2 mm
bore Teflon stopcock.

Mini Vials, 5 ml, with Teflon-lined caps--Available from Alltech Associates, 202 Campus Drive, Arlington Heights, Illinois 60004.

Oven--Forced draft, with settings of 80° and 130°C.

Polyethylene Sheet--3 mil, for Blender-jar cap liners.

Reflux Condenser--with $\frac{1}{8}$ 24/40 joint, 300 mm Allihn or similar.

Rotary Vacuum Evaporator-- $\frac{1}{8}$ 24/40 joint, adaptor for Bantamware joint, $\frac{1}{8}$ 24/40 to 14/20.

Round-Bottom Flasks--With $\frac{1}{8}$ 24/40 joint, 300 ml, 500 ml, 1000 ml.

Rubber Stoppers, No. 6--bored to fit the Chromatographic Column as illustrated in Fig. 1.

Separatory Funnel--250 ml.

Serological Pipettes--1 ml, 2 ml, 5 ml, 10 ml.

Shaker--Bench-type, lateral action, variable speed. Available from A. H. Thomas.

Spatula--Hayman microspatula. A. H. Thomas Cat. No. 8340-H20.

Stirring Hot Plate--Corning Model PC-361 or equivalent.

Transfer Pipettes--Various sizes including a 4 ml Class A.

"Titrats"--Automatic measuring with $\frac{1}{4}$ 24/40 joint. 5 ml, 10 ml, 15 ml, 25 ml, 50 ml, 75 ml, 100 ml.

Volumetric Flasks--100 ml, 250 ml, 500 ml, 1000 ml.

Watch Glass--At least 90 mm in diameter.

Water Bath--Capable of adjustment within a range of 55° to 60°C.

Reagents

Acetone--Burdick & Jackson "Distilled-in-Glass" grade.

Aluminum Oxide--Baker "Analyzed" Reagent Grade or Fisher A-540

Adsorption Alumina. Wash each pound of this reagent with 1 liter of anhydrous methanol, filter with vacuum on No. 1 filter paper, wash again with 100 ml of methanol, allow to air dry, activate at 130°C for 2 to 3 hours, and store in a tightly capped container in a desiccator.

Benzene--Burdick & Jackson "Distilled-in-Glass" grade.

1-Bromobutane--Matheson, Coleman & Bell, Cat. No. BX 1045. Purify by passing 100 ml of the reagent through a 2.2 cm-diameter x 10 cm-long chromatographic column containing 40 grams of activated aluminum oxide and capped with a glass-wool plug. Do this operation in a hood.

Dimethylformamide (DMF)--Burdick & Jackson "Distilled-in-Glass" grade.

Ethylenethiourea (ETU)--Eastman Practical Grade, Cat. No. B 5950.

Toxic; see "Safety" section.

Methanol--Burdick & Jackson "Distilled-in-Glass" grade.

Hydrochloric Acid--Concentrated, reagent grade.

~~Sea~~ Sand, washed and ignited--Fisher Scientific Co.

Sodium Borohydride (NaBH_4)--Fisher Scientific Co., Cat. No. S-678 (10 grams). This material is hygroscopic; order the smallest available containers, and avoid prolonged exposure to air. Store in a desiccator.

Sodium Hydroxide Solution, 20%--Weigh 50 grams of reagent-grade sodium hydroxide pellets into a 300 ml beaker, add 200 ml of distilled water, and stir to dissolve. Store in a capped, labelled, polyethylene container.

Wash Solution: 5% Acetone in Benzene--Prepared by mixing 50 ml of acetone with 950 ml of benzene.

Elution solvent: 7% Methanol in Wash Solution--Prepared by mixing 70 ml of methanol with 930 ml of Wash Solution.

Petroleum Ether--Burdick & Jackson "Distilled-in-Glass" grade.

Hexane--Burdick & Jackson "Distilled-in-Glass" grade.

Methanol/Water Solution, 50/50--prepare in convenient quantities by mixing equal volumes of methanol and distilled water.

(Extraction Solvent--methanol, acetone, water, trichloroacetic acid--(250:127:137.5:1V/V)*)

Special Reagents

Recrystallized ETU - Preparation:

Transfer 100 ml of methanol/water solution (50/50) into a 250 ml Erlenmeyer flask. Add 5 grams of ETU to the flask and heat the contents on a hot plate, swirling intermittently, until the ETU has dissolved. Place the flask in a pan of running tap water to cool for five minutes.

*As modified by WFP/MDG

After cooling, add 5 ml of hexane, shake the flask vigorously, and transfer it to a freezer. At the same time transfer a container with 100 ml of methanol/water to the freezer. Allow the ETU to recrystallize overnight. Filter the ETU crystals using a fine-porosity filter funnel under vacuum. Rinse the crystals with two 15 ml portions of chilled methanol/water solution. Allow the crystals to air dry overnight, then, transfer them to a watch glass, and finish drying them in an 80°C oven for three hours. Determine the melting point range of the dried crystals; if the melting range exceeds 1.2°C, or if the initial softening occurs below 198°C, repeat the recrystallization using the dried crystals. Store the finished, recrystallized ETU in a desiccator.

S-Butyl Derivative - Preparation:

Transfer 2 grams of recrystallized ETU to a 500 ml Erlenmeyer flask with a 24/40 joint. Separately mix 20 ml of 1-bromobutane with 180 ml of methanol; add this mixture, plus 50 ml of 20% sodium hydroxide, and a magnetic stirring bar to the contents of the Erlenmeyer flask. Connect a reflux condenser to the flask, and place it on a stirring hot plate. Stir the contents and heat them until the solution refluxes at a rate of 1 to 3 drops per second. Allow the refluxing to continue, adjusting the hot plate setting as necessary to maintain this rate, for 10 minutes. Allow the flask contents to cool, and transfer them to a 500 ml separatory funnel. Add 100 ml of benzene, and shake the funnel vigorously for about two minutes, venting the stopcock frequently to avoid pressure increase. Allow the layers to separate; drain the lower layer into another separatory funnel, and transfer the upper layer to a clean

500 ml Erlenmeyer flask. Repeat the extraction with three additional 100 ml portions of benzene, adding the upper layers of each extraction to the same 500 ml Erlenmeyer flask.

Transfer the benzene solution to a 2.2 cm chromatographic column containing a 5 cm bed of anhydrous sodium sulfate; collect the column effluent in a 1000 ml, round-bottom flask with $\frac{1}{2}$ 24/40 joint. After all the benzene has passed through the column, wash the column with two 20 ml portions of benzene, adding this effluent to the round-bottom flask. Connect this flask to a rotary evaporator, slowly evacuating the flask while rotating it, and immerse it in a water bath adjusted to $65 \pm 5^\circ\text{C}$. Concentrate the solution to between 60 and 80 ml, transfer it to a 125 ml, round-bottom flask with $\frac{1}{2}$ 24/40 joint, and continue to concentrate the solution until between 4 and 5 ml remain in the flask.

Remove the flask from the evaporator, and add 25 ml of water. Swirl to dissolve the material, and transfer it to a 125 ml separatory funnel. Add 25 ml of petroleum ether to the funnel, and shake vigorously. Allow the layers to separate, and transfer the lower (water) layer to another 125 ml separatory funnel. Pour the upper (petroleum ether) layer into a 250 ml Erlenmeyer flask. Repeat the extraction of water with two more 25 ml portions of petroleum ether, adding these upper layers to the 250 ml Erlenmeyer flask. Transfer the solution from the Erlenmeyer flask to a chromatographic column containing a fresh, 5 cm bed of anhydrous sodium sulfate. Rinse the Erlenmeyer flask with 25 ml of petroleum ether, and when the liquid in the column reaches the top of the packing, transfer the rinse to the column. Collect the column effluent in a 300 ml round-bottom flask with $\frac{1}{2}$ 24/40 joint.

Adjust the water-bath temperature to 55°C. Connect the 300 ml flask to the rotary evaporator, immerse the flask in the bath, and evaporate the petroleum ether without vacuum until only a few milliliters of liquid remain. Apply vacuum to the flask for only a few seconds, to remove the remaining petroleum ether. Place the flask in the freezer overnight, then decant the supernatant liquid from the crystals, transfer them to a sheet of filter paper, and allow them to air dry overnight. Transfer the crystals to a 50 ml beaker and add about 10 ml of petroleum ether; warm the beaker on a steam bath, and stir to dissolve the crystals. Continue heating until most of the ether has evaporated. Transfer the moist crystals to a clean sheet of filter paper on a watch glass, and place the glass in the freezer until crystallization is complete. Transfer the crystals to another 50 ml beaker, and repeat the recrystallization from 10 ml of petroleum ether. Store the recrystallized product in the freezer in a capped, labelled container.

ETU Spiking Solutions - Preparation:

Weigh 0.0250 gram of recrystallized ETU into a 500 ml volumetric flask. Dissolve and dilute to the graduation mark with distilled water, and mix well. Label this Solution "A-1" (50 µg/ml ETU) and store in a capped, amber bottle under refrigeration; allow this solution to warm to room temperature before using. Prepare Working Solution "A-2" (1 µg/ml ETU): pipette 5.0 ml of Solution "A-1" into a 250 ml volumetric flask, dilute to the graduation mark with distilled water, and mix well. Prepare Working Solution "A-3" (0.40 µg/ml ETU): pipette 100 ml of Solution "A-2" into a 250 ml volumetric flask, dilute to the graduation

mark with distilled water, and mix well. Store Working Solutions "A-2" and "A-3" in capped, labelled, amber bottles at room temperature; retain these solutions no longer than 3 months.

Standard Solutions - Preparation:

Weigh 0.0250 gram of recrystallized S-Butyl ETU derivative into a 250 ml volumetric flask. Dissolve and dilute to the graduation mark with benzene, and mix well. Label this Solution "B-1" (100 $\mu\text{g/ml}$ of derivative) and store in a capped, amber bottle under refrigeration; allow the solution to warm to room temperature before using. Prepare Solution "B-2" (10 $\mu\text{g/ml}$ of derivative which is equivalent to 6.46 $\mu\text{g/ml}$ as ETU): pipette 10.0 ml of Solution "B-1" into a 100 ml volumetric flask, dilute to the graduation mark with benzene, and mix well. Solution "B-2" is used to prepare the eight Working Standard Solutions in the following Table I. Using a 50 ml burette (Certified, NBS), transfer the indicated volumes of Solution "B-2" into 8 respective 100 ml volumetric flasks, dilute to the graduation mark with benzene, and mix well. Store these solutions in tightly capped, labelled, amber bottles at room temperature, and retain no longer than 4 months. Discard the remaining Solution "B-2".

Table I

<u>Volume of Solution</u> <u>"B-2" in ml</u>	<u>Micrograms/Milliliter</u> <u>as ETU</u>
3.1	0.2
6.2	0.4
7.6	0.5
9.3	0.6
10.9	0.7
12.4	0.8
14.0	0.9
15.6	1.0

Procedure

A set of analyses requires at least 120 grams of a Control (untreated) sample and 40 grams of each treated sample. By the term "treated" we mean material which has been exposed to Dithane.

Because the procedure is lengthy, and because results may be decreased by time delays during certain critical operations, a single operator should not normally attempt to run more than five treated samples concurrently. This analysis normally requires two days; the first for preparation of the samples for gas chromatography, and the second for the chromatographic analyses.

Determine the volumes of ETU Spiking Solution (A-2 or A-3) required for two "fortification" levels: one, approximately equal to the expected ETU content of the treated samples, which is determined according to the

following calculation; and the second twice the first. (NOTE: If no detectable ETU content is expected in the treated samples, use fortification levels of 0.02 and 0.04 ppm.)

(1) Volume of ETU Spiking Solution =

$$\frac{(\text{Expected Sample ETU, ppm}) \times 40 \text{ g Sample}}{\text{Spiking Solution Concentration, } \mu\text{g/ml}}$$

For example:

$$\text{Volume} = \frac{0.05 \text{ ppm} \times 40 \text{ g}}{1 \text{ } \mu\text{g/ml}} = 2 \text{ ml of Spiking Solution A-2}$$

and, Volume of second Spike = 4 ml of Spiking Solution A-2.

Pipette the calculated fortification volumes of Spiking Solution A-2 or A-3 into Control samples when indicated by the procedure.

Sample Preparation

Carry each sample (one Control, two Controls for fortification, and each treated sample) consecutively through the procedure in the following two paragraphs, to the indicated point.

Weigh 40 grams $\pm$ 0.1 g of sample (use a top-loading balance) directly into a 1-pint, glass Waring Blender jar. Add [167 ml of the extraction mixture]^{*} to the jar. (NOTE: Add the required volume of Spiking Solution to each of the two Controls reserved for fortification; these two samples will now be referred to as "Spiked Controls".) Cover the jar with a piece of polyethylene sheet, and tighten the metal screw cap over it. Blend the mixture at low speed for two minutes. Transfer the blended mixture quantitatively to No. 541 Whatman filter paper in a Buchner funnel; rinse the plastic liner (into the funnel) with methanol, and rinse the Blender jar twice with 30 ml portions of methanol, adding the

^{*}As modified by Technological Resources Inc.

rinses to the funnel. Filter the mixture (with vacuum) into a 1000 ml filter flask, rinsing the inside walls of the funnel and the filter cake with about 25 ml of methanol. Transfer the filtrate quantitatively to a 500 ml round-bottom flask, rinsing the filter flask with small portions of methanol (limit the total solution volume to about 300 ml). Mark each round-bottom flask with the identity of its contents. (NOTE: Bring all samples to this point before proceeding.)

Set the water bath temperature to 80°C. Connect the round-bottom flasks (in groups of four) to the rotary evaporator (condenser stopcocks in the open position) and immerse them in the water bath. Allow the contents of each flask to distill (without vacuum) into the discard trap; when the refluxing stops, close the condenser stopcock, and apply vacuum. Concentrate the contents of the flask until approximately 3 to 5 ml remain; then remove the flask from the evaporator and immediately transfer its contents (use a 4 ml disposable, capillary pipette) to a 10 ml glass-stoppered graduated cylinder. Rinse the round-bottom flask with two equal (1 to 2 ml) portions of distilled water and add both rinses to the cylinder (Do not exceed 6 ml total volume in the cylinder). Dilute the graduated cylinder to 6 ml with distilled water, stopper, and mix well. (NOTE: Bring all samples to this point before proceeding to the next section.)

Liquid Chromatographic Purification

NOTE: Complete all the following operations (up to the acidification step in the "Derivative Formation" section) as quickly as possible; ETU in the sample decomposes rapidly, especially in DMF solutions.

Prepare a chromatographic column for each sample as shown in Figure 1. Place a 250 ml vacuum filtration flask under the column outlet and secure it tightly to the rubber stopper. NOTE: The column stopcock remains fully open during all the following operations. Using a serological pipette, transfer 1.0 ml of the aqueous extract from the 10 ml cylinder directly to the top of the column packing. Add two 1 ml portions of benzene to rinse the pipette and allow each of these rinses to drain into the top of the column, about 1/4 inch below the surface of the sand; then add 25 ml of benzene to the column. Allow the benzene to fill the column by gravity, then apply vacuum to the filter flask (the filtration rate is adjusted to about (4)^{*} ml/minute with a "C" clamp which is attached to the vacuum line). NOTE: To this point, do not allow the liquid level to fall below the column-packing surface during any step; the packing will crack and channel if this happens. When the liquid level at the top of the column falls to the sand surface, add 75 ml of "Wash Solution" (5% acetone/benzene); then when the liquid level at the top of the column falls to the sand surface, disconnect the vacuum line, discard the filtrate, and connect a fresh filtration flask (250 ml) to the column outlet. Add (90)^{*} of "Elution Solvent" (7% methanol in Wash Solution) to the column and re-apply vacuum at the same 12 ml/minute rate. Allow the liquid to drain entirely from the column into the flask; then transfer the eluate to a 300 ml round-bottom flask. Rinse the filtration flask with 25 ml of methanol and add the rinse to the round-bottom flask. Using a Titet dispenser, add 10.0 ml of distilled water and 5.0 ml of dimethylformamide (DMF) to the round-bottom flask.

^{*}As modified by Technological Resources Inc.

Connect the flask to the rotary evaporator and immerse the flask in a water bath adjusted to $78 \pm 3^\circ\text{C}$. Allow the more volatile solvents (acetone and methanol) to evaporate; then apply vacuum and evaporate the solution until the volume is reduced to between two and three milliliters. Immediately transfer this solution to a 10 ml glass stoppered graduated cylinder, using a 2 ml capillary dropping pipette. Rinse the flask twice with 1 ml portions of DMF, adding both rinses to the cylinder. Dilute the cylinder to 6.0 ml with DMF.

Derivative Formation

Using a 1 ml serological pipette, add 0.3 ml of 1-bromobutane to the graduated cylinder and swirl to mix. Fill the scoop end of a Hayman microspatula one-third full with sodium borohydride (about 0.01 g), and add this reagent to the cylinder; stopper and shake vigorously for about 10 seconds. Pipette 1.0 ml of 20% sodium hydroxide solution into the cylinder and shake it vigorously for 10 seconds, then allow it to stand at room temperature for 5.0 minutes.

Transfer 4.0 ml (by transfer pipette) of the upper layer from the cylinder to a 50 ml Bantamware Erlenmeyer flask. Add 1 drop of concentrated hydrochloric acid, connect the flask through an adaptor to the rotary evaporator, and mark the flask with the identity of its contents.

(NOTE: After the acid addition time is no longer critical.) Immerse the flask in a water bath adjusted to $68 \pm 3^\circ\text{C}$, apply vacuum and allow the solution to evaporate to dryness. At this point, flasks containing dried derivative may be removed from the evaporator, cooled, stoppered, and retained until their gas chromatographic analysis is completed.

Gas Chromatographic Analysis

Treat each dried derivative from the preceding section according to the following procedure. Add 0.5 ml of distilled water to the Bantamware flask, and swirl until the contents are dissolved. Add 0.5 ml of 20% sodium hydroxide solution and 0.5 ml of benzene; stopper tightly, shake the mixture for one minute, and allow it to stand for five minutes. Pour the flask contents into a 5 ml Mini Vial, place the Vial in a centrifuge, and counterbalance it. Centrifuge the Vial for 15 minutes at approximately 1700 - 1800 rpm.

Set the gas chromatograph to the following conditions, and allow it to stabilize:

Column	6 ft x 1/4 in, (10% Carbowax 20M or Carbowax 20M Terephthalic acid + 5% KOH)* on Gas Chrom Q
Column Temperature	(195°C)* Isothermal
Detector Temperature	(180°C)*
Injection Port Temperature	(205°C)*
Carrier Gas	Nitrogen at 55 ml/minute
Air Flow	(30)* ml/minute
Oxygen Flow	10 ml/minute
Hydrogen Flow	(70)* ml/minute
Photomultiplier Voltage	750 Volts
Attenuation	Variable (approximately 64×10)*

*As modified by Technological Resources Inc.

The following injection volume constraints apply to all samples:

1. Single injections are not to exceed 25 μ l.
2. Injection volumes within the range 10 μ l to 25 μ l vary by increments of 5 μ l.
3. Injection volumes within the range 1 μ l to 10 μ l vary by increments of 1 μ l.

Trial Injections:

Inject 10 μ l of the 0.8 μ g/ml Standard Solution into the instrument, and allow the ETU derivative peak to elute (Figure 2). Similarly inject 10 μ l of the 0.2 μ g/ml Standard; measure and record the peak heights, in chart units, of each ETU derivative peak. The 0.8 μ g/ml Standard must produce a peak with about 70% chart deflection and the 0.2 μ g/ml Standard must produce a measurable peak; if either of these criteria cannot be met, refer to the chromatographic instrument manual to correct low-sensitivity problems and repeat the trial injections. From this point on, sample peak height adjustments (to maintain on-scale chromatograms) are made by varying the sample injection volumes only. Make no attenuation adjustments.

Control Sample:

Inject 25 μ l of the Control sample (from the upper layer in the appropriate Mini Vial); allow the ETU derivative peak to elute, measure and record the peak height (in chart units), and record the injection volume (NOTE: If the ETU derivative peak is off scale with any injection, reduce the injection volume until the peak remains on scale, and record the injection volume used).

Spiked Control Samples:

Determine and record the μg ETU added to the Spiked Control samples using the following equation:

$$(2) \text{ } \mu\text{g ETU Added} = \left[\begin{array}{l} \text{Volume of ETU Spiking} \\ \text{Solution Added, ml} \\ \text{(from Calculation 1)} \end{array} \right] \times \left[\begin{array}{l} \text{Concentration of Spiking} \\ \text{Solution, } \mu\text{g/ml (Working)} \\ \text{Solution A-2 or A-3} \end{array} \right]$$

Estimate an injection volume for the higher of the two Spiked Control samples from the following equation:

$$(3) \text{ Higher Spiked Control Injection Volume for} = \frac{(\text{Std. Sol'n from Trial Injection}) (\text{Dilution Factor})}{\text{ug ETU Added to the Higher Spiked Control (from Calculation 2)}}$$

$\frac{0.8 \text{ } \mu\text{g/ml} \times 10 \text{ } \mu\text{l}}{6.96 \text{ ml}}$

Estimate an injection volume for the Lower Spiked Control by doubling the injection volume of equation (3). NOTE: These estimated injection volumes are guides and may require adjustment because of instrument sensitivity variation. Record the injection volumes used for each Spiked Control.

Make duplicate injections of the selected volume of each Spiked Control sample (Mini Vial upper layer) into the chromatographic column, allowing the ETU derivative peak to elute from the previous injection before making the next injection. Measure and record the ETU derivative peak heights, in chart units.

Standard Samples:

Run a series of three Standard samples to establish a Calibration Curve. Adjust the injection volumes of these standards so that they produce peak heights within the range of about 5 to 70% chart deflection. Measure and record the peak heights of each ETU derivative peak, in chart units.

Treated Samples:

Inject 25 μ l of the Treated sample (Mini Vial upper layer) into the chromatographic column, allow the ETU derivative peak to elute, measure ~~and~~ record the peak height and injection volume. If the ETU derivative peak height exceeds the peak height of the highest Standard used, reduce the injection volume until re-injection produces a derivative peak height within the range of the Standards (5 to 70% chart deflection).

Similarly inject the remaining Treated samples; read and record the peak heights and injection volumes used. After approximately 10 samples, re-inject the High Standard derivative solution, and after running the last Treated sample, again repeat the High Standard injection. Measure and record the ETU derivative peak heights.

Determine the ETU content of the S-butyl ETU derivative Standards using the following calculation:

$$(4) \text{ Nanograms (ng) ETU} = \left[\frac{\text{Injection Volume of Standard}}{\mu\text{l}} \right] \times \left[\frac{\text{Concentration of Standard, } \mu\text{g./ml}}{(\text{as ETU})} \right]$$

On a sheet of 10 x 10 per centimeter graph paper plot the peak height of each ETU derivative Standard versus its corresponding ETU content (nanograms of ETU from Calculation 4). Draw the line that most closely fits the set of points; this is the ETU calibration curve.

Using the calibration curve, determine the nanograms of ETU corresponding to the peak heights for the two Spiked Control samples, and the untreated Control sample. Record these values as "nanograms ETU from Curve"; they represent the nanograms of ETU in the gas chromatographic

injection. Determine the "total micrograms of ETU found" using the following calculation which corrects for dilution and unit conversion; these values represent the total micrograms of ETU in the original sample:

$$(5a) \text{ Total } \mu\text{g ETU Found} = \text{ng ETU from Curve} \times \frac{0.5 \text{ ml in Mini Vial}}{\mu\text{l Injected in GLC}} \times \frac{6.95 \text{ ml}^*}{4.0 \text{ ml Aliquot}} \times \frac{8.0 \text{ ml}}{1.0 \text{ ml Aliquot}} \times \frac{1000 \mu\text{l/ml}}{1000 \text{ ng}/\mu\text{g}}$$

or

$$(5b) \text{ Total } \mu\text{g ETU Found} = \frac{(\text{ng ETU from Curve}) \times 6.95}{\mu\text{l Injected into GLC}}$$

*6.95 ml is the estimated volume of the organic phase in the 10 ml graduated cylinder before the 4.0 ml aliquot portion is pipetted from it.

Determine and record the "micrograms of net ETU, Recovered" for each Spiked Control sample using the following calculation; these values represent the total micrograms of ETU found in the Spiked samples, corrected for the total micrograms of ETU contributed by the untreated Control sample:

$$(6) \mu\text{g of net ETU, Recovered} = \left[\text{Total } \mu\text{g ETU Found for Spiked Control} \right] - \left[\text{Total } \mu\text{g ETU Found for untreated Control}^* \right]$$

*If this value is less than 0.0 μg , ignore it and report the Total μg ETU Found for the Spiked Control as the μg of net ETU, Recovered.

Determine and record the "percent recovery" for each Spiked Control sample, using the following calculation; these values represent the amount of ETU found in each Spiked Control, expressed as a percentage of the amount of ETU actually added:

$$(7) \% \text{ Recovery} = \frac{\mu\text{g of net ETU, Recovered (from calculation 6)}}{\mu\text{g ETU added (from calculation 2)}} \times 100$$

The percent recovery must be 60% or greater for one or more Spiked Controls (except at very low levels); if it is not, repeat the entire determination of the Control sample, Spiked Controls, and Treated Samples. If the percent recovery for both Spiked Controls is 60% or greater, determine the average of the two values; if only one value is 60% or greater, use that value in later calculations, and disregard the value below 60%.

Using the calibration curve, determine and record the nanograms of ETU corresponding to the peak heights for the treated samples. Determine and record the "total" micrograms of ETU found in the treated samples, using the following calculation:

$$(8a) \text{ Total } \mu\text{g ETU Found} = \frac{(\text{ng ETU from Curve}) \times 6.96}{\mu\text{l Injected into GLC}}$$

Correct the Total μg of ETU Found, for the % Recovery, using the following calculation:

$$(8) \mu\text{g ETU Corrected} = \frac{\text{Total } \mu\text{g ETU Found} \times 100}{\text{Average } \% \text{ Recovery}}$$

Determine and record the "parts per million ETU" in the Treated Samples, using the $\mu\text{g ETU Corrected}$, and the following calculation:

$$(9) \text{ ppm ETU} = \frac{\mu\text{g ETU Corrected (from Calculation 8)}}{\text{Sample Weight in grams}}$$

(Attached 3 Figures)

Figure 1. Preparation of liquid chromatography column

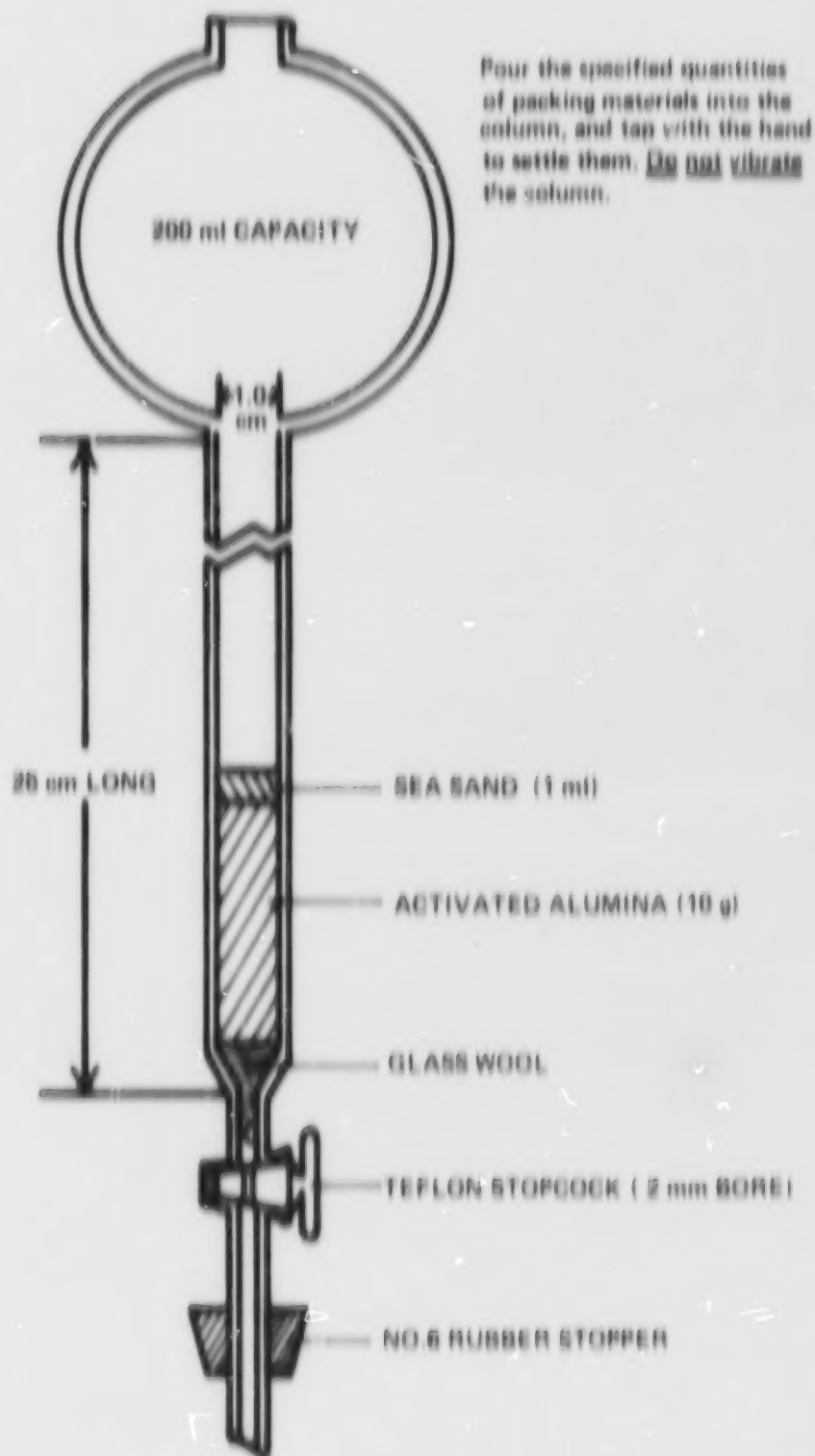


Figure 2. Typical chromatograms (ETU)

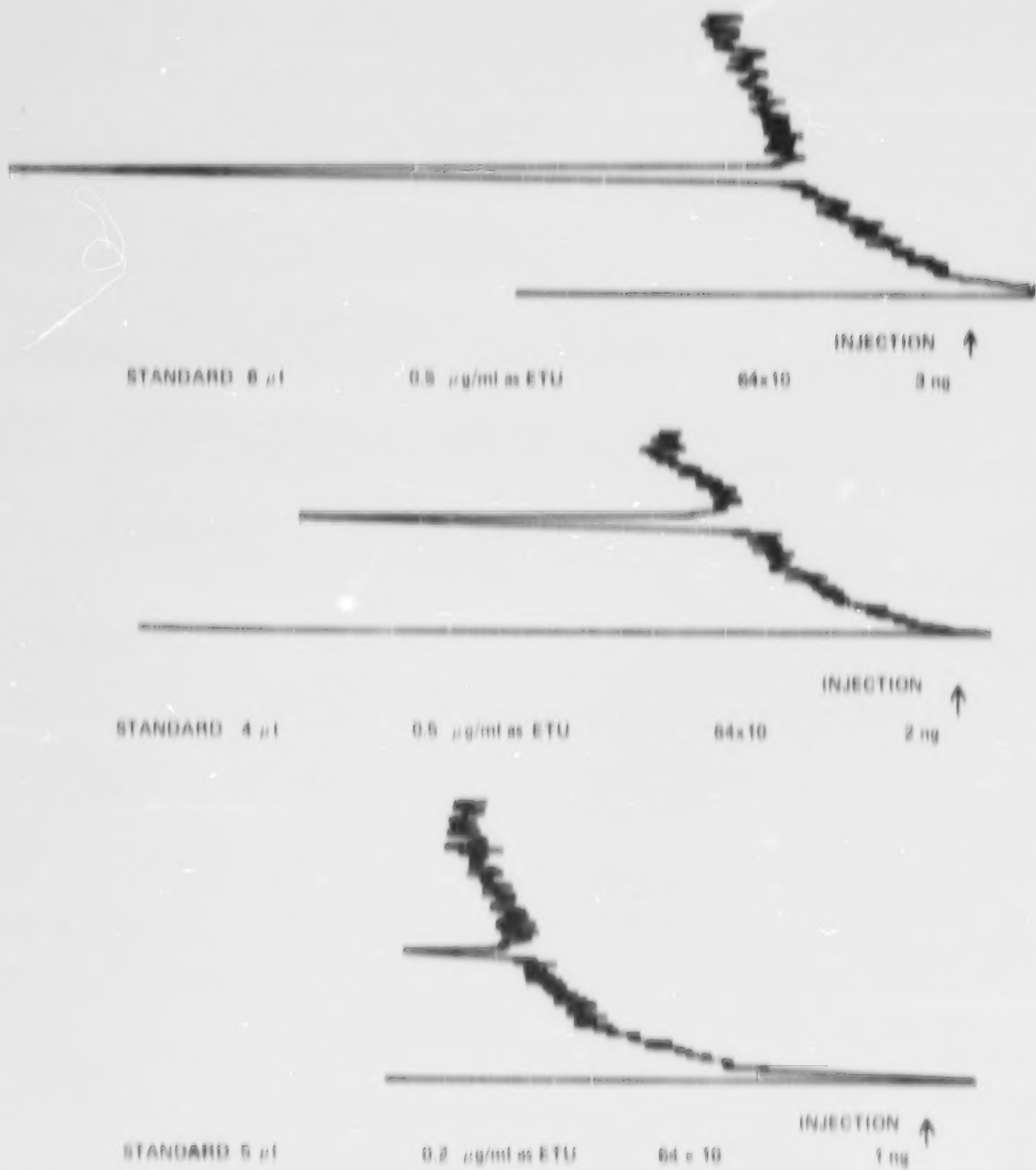
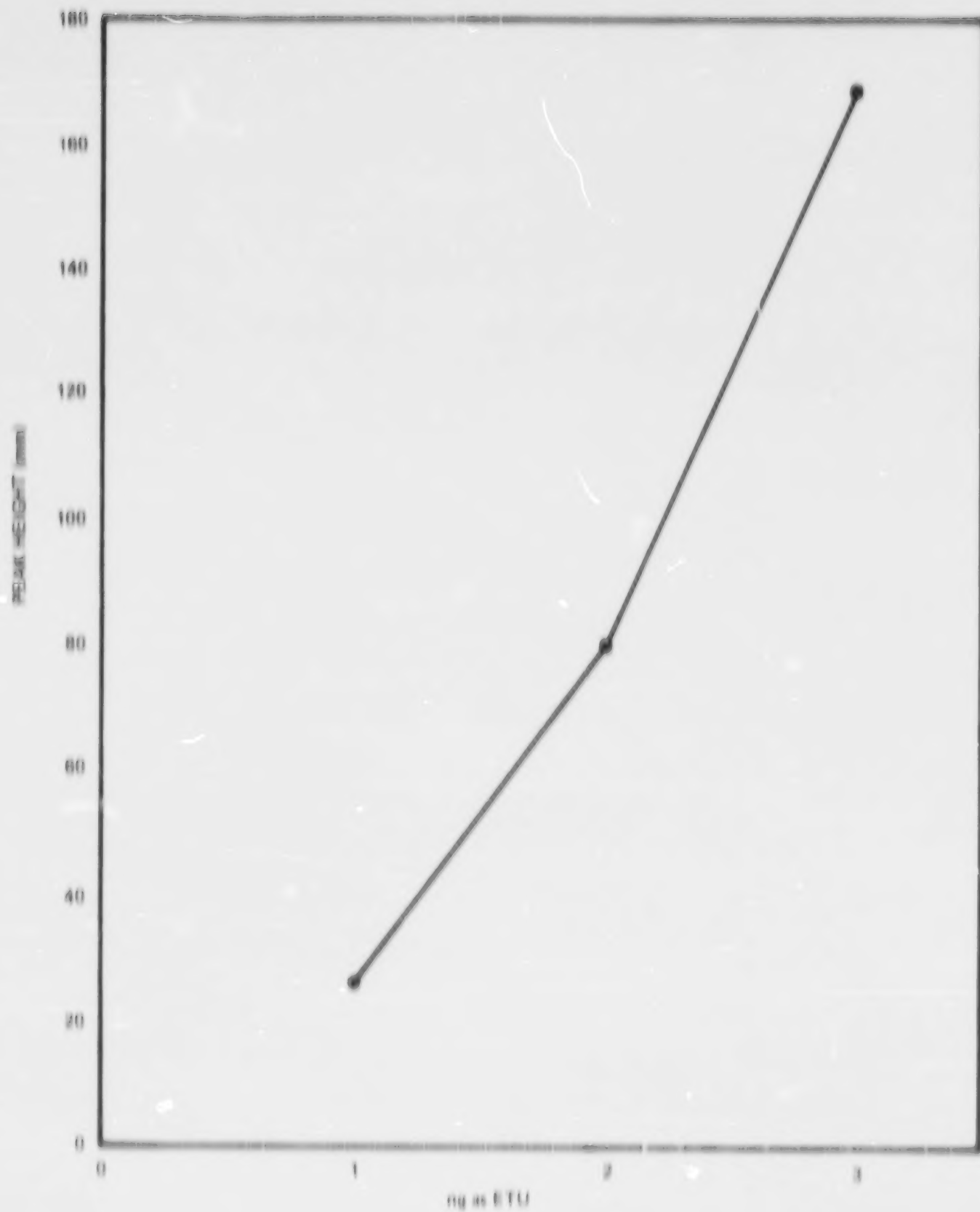


Figure 3. Typical calibration curve



APPENDIX B

INDIVIDUAL DATA FROM SUMMARY TABLE 3

Residues of EBDC and ETU (from Dithane M-45[®])
in Tomatoes, Carrots, and Spinach at Process Stages

Table 1A-1-A. Residues of EBDC (from M-46) in
East Coast Unwashed Tomatoes

Sample	Amount Found EBDC (ppm)	Average	*Corrected for Check	Average % Recovery	Corrected for Avg. % Recovery
Rep I check	<0.1, <0.1	<0.1			
Rep II check	<0.1, <0.1	<0.1			
Rep III check	<0.1, <0.1	<0.1			
Rep I 1x	<0.1, 0.1	0.1	0.1	108	0.1
Rep II 1x	0.2, 0.2	0.2	0.2		0.2
Rep III 1x	0.6, 0.6	0.6	0.6		0.6
Rep I 4x	0.9, 0.9	0.9	0.9	108	0.9
Rep II 4x	0.7, 0.6	0.7	0.7		0.6
Rep III 4x	0.8, 0.8	0.8	0.8		0.7

Recovery Studies EBDC (ppm)

	Amount Added	Amount Found	*Corrected for Check	% Recovery	Average % Recovery
Rep I check	1.2 EBDC**	1.2, 1.2	1.2, 1.2	100, 100	
Rep II check	1.2 EBDC**	1.7	1.7	142	
Rep III check	1.2 EBDC**	1.0, 1.1	1.0, 1.1	83, 92	
Rep I check	2.9 EBDC**	3.1, 3.3	3.1, 3.3	107, 114	
Rep II check	2.9 EBDC**	4.2	4.2	145	
Rep III check	2.9 EBDC**	2.7	2.7	93	108

*Corrected by subtraction of amount found in check sample.

**Amount added has been corrected for % activity of 57.6.

Table 1A-1-B. Residues of EBDC (from M-45) in
East Coast Washed Tomatoes

Sample	Amount Found EBDC (ppm)	Average	*Corrected for Check	Average % Recovery	Corrected for Avg. % Recovery
Rep I check	<0.1, <0.1	<0.1			
Rep II check	0.1, 0.1	0.1			
Rep III check	0.2, 0.2	0.2			
Rep I 1x	0.3, 0.3	0.3	0.3	97.3	0.3
Rep II 1x	0.3, 0.2	0.3	0.2		0.2
Rep III 1x	0.2, 0.2	0.2	<0.1		<0.1
Rep I 4x	0.4, 0.4	0.4	0.4	97.3	0.4
Rep II 4x	0.4, 0.4	0.4	0.3		0.3
Rep III 4x	0.4, 0.4	0.4	0.2		0.2

Recovery Studies EBDC (ppm)

	Amount Added	Amount Found	*Corrected for Check	% Recovery	Average % Recovery
Rep I check	1.2 EBDC**	1.1, 1.2	1.1, 1.2	92, 100	
Rep II check	1.2 EBDC**	1.0, 1.1	0.9, 1.0	75, 83	
Rep III check	1.2 EBDC**	1.6, 1.6	1.4, 1.4	117, 117	
Rep I check	2.9 EBDC**	2.7, 2.9	2.7, 2.9	93, 100	
Rep II check	2.9 EBDC**	2.4, 2.7	2.3, 2.6	79, 90	
Rep III check	2.9 EBDC**	3.5, 3.6	3.3, 3.6	114, 124	97.1

*Corrected by subtraction of amount found in check sample.

**Amount added has been corrected for % activity of 57.6.

Table IA-1-G. Residues of EBDC (from M-45) in
East Coast Tomato Tailings

Sample	Amount Found EBDC (ppm)	Average	*Corrected for Check	Average % Recovery	Corrected for Avg. % Recovery
Rep I check	<0.1, <0.1	<0.1			
Rep II check	<0.1, <0.1	<0.1			
Rep III check	0.1, 0.2	0.2			
Rep I 1x	0.1, 0.1	0.1	0.1	94.7	0.1
Rep II 1x	0.2, 0.3	0.3	0.3		0.3
Rep III 1x	0.3, 0.3	0.3	0.1		0.1
Rep I 4x	0.6, 0.7	0.7	0.7	94.7	0.7
Rep II 4x	0.6, 0.6	0.6	0.6		0.6
Rep III 4x	0.4, 0.6	0.6	0.3		0.3

Recovery Studies EBDC (ppm)

	Amount Added	Amount Found	*Corrected for Check	% Recovery	Average % Recovery
Rep I check	1.2 EBDC**	1.0	1.0	83	
Rep II check	1.2 EBDC**	1.0	1.0	83	
Rep III check	1.2 EBDC**	1.2	1.0	83	
Rep I check	2.9 EBDC**	3.1	3.1	106	
Rep II check	2.9 EBDC**	3.0	3.0	103	
Rep III check	2.9 EBDC**	3.4	3.2	110	94.7

*Corrected by subtraction of amount found in check sample.

**Amount added has been corrected for % activity of 97.6.

Table IA-1-D. Residues of EBDC (from H-45) in
Canned Juice from East Coast Tomatoes

Sample	Amount Found EBDC (ppm)	Average	*Corrected for Check	Average % Recovery	Corrected for Avg. % Recovery
Rep I check	0.1, 0.1	0.1			
Rep II check	0.1, 0.1	0.1			
Rep III check	<0.1, 0.1	0.1			
Rep I 1x	<0.1, <0.1, 0.1, 0.2	0.1	<0.1	90.1	<0.1
Rep II 1x	<0.1, <0.1	<0.1	<0.1		<0.1
Rep III 1x	<0.1, <0.1	<0.1	<0.1		<0.1
Rep I 4x	0.2, 0.3	0.3	0.2	90.1	0.1
Rep II 4x	0.2, 0.2	0.2	0.1		0.1
Rep III 4x	0.1, 0.1	0.1	<0.1		0.1

Recovery Studies EBDC (ppm)

	Amount Added	Amount Found	*Corrected for Check	% Recovery	Average % Recovery
Rep I check	1.2 EBDC**	1.0, 1.1	0.9, 1.0	75, 83	
Rep II check	1.2 EBDC**	0.9, 1.1	0.8, 1.0	67, 83	
Rep III check	1.2 EBDC**	1.2, 1.4	1.1, 1.3	92, 108	
Rep I check	2.9 EBDC**	2.6, 2.8	2.6, 2.7	86, 93	
Rep II check	2.9 EBDC**	2.6, 3.0	2.6, 2.8	90, 97	
Rep III check	2.9 EBDC**	3.0, 3.2	2.9, 3.1	100, 107	90.1

*Corrected by subtraction of amount found in check sample.

**Amount added has been corrected for % activity of 57.6.

Table 1A-2-A. Residues of ETU (from M-46) in
East Coast Unwashed Tomatoes

Sample	Amount Found ETU (ppm)	Average	*Corrected for Check	Average % Recovery	Corrected for Average % Recovery
Rep I check	<0.01, <0.01	<0.01			
Rep II check	<0.01, <0.01	<0.01			
Rep III check	<0.01, <0.01	<0.01			
Rep I 1x	<0.01, <0.01	<0.01	<0.01	86.7	<0.01
Rep II 1x	0.01, 0.01	0.01	0.01		0.01
Rep III 1x	<0.01, <0.01	<0.01	<0.01		<0.01
Rep I 4x	0.01, 0.02	0.02	0.02	86.7	0.02
Rep II 4x	0.01, 0.02	0.02	0.02		0.02
Rep III 4x	0.02, 0.02	0.02	0.02		0.02

Recovery Studies ETU (ppm)

	Amount Added	Amount Found	*Corrected for Check	% Recovery	Average % Recovery
Rep I check	0.05 ETU	0.07	0.07	140	
Rep II check	0.05 ETU	0.03	0.03	60	
Rep III check	0.05 ETU	0.03	0.03	60	86.7

*Corrected by subtraction of amount found in check sample.

Table IA-2-B. Residues of ETU (from M-45) in
East Coast Washed Tomatoes

<u>Sample</u>	<u>Amount Found ETU (ppm)</u>	<u>Average</u>	<u>*Corrected For Check</u>	<u>Average % Recovery</u>	<u>Corrected for Average % Recovery</u>
Rep I check	0.01, 0.01	0.01			
Rep II check	0.01, 0.01	0.01			
Rep III check	0.01, 0.01	0.01			
Rep I 1x	0.02, 0.01	0.02	0.01	80	0.01
Rep II 1x	<0.01, <0.01	<0.01	<0.01		<0.01
Rep III 1x	0.01, 0.01	0.01	<0.01		<0.01
Rep I 4x	0.01, 0.01	0.01	<0.01	80	<0.01
Rep II 4x	0.02, 0.02	0.02	0.01		0.01
Rep III 4x	0.02, 0.02	0.02	0.01		0.01

Recovery Studies ETU (ppm)

	<u>Amount Added</u>	<u>Amount Found</u>	<u>*Corrected For Check</u>	<u>% Recovery</u>	<u>Average % Recovery</u>
Rep I check	0.05 ETU	0.05	0.05	100	
Rep II check	0.05 ETU	0.05	0.04	80	
Rep III check	0.05 ETU	0.04	0.03	60	80

*Corrected by subtraction of amount found in check sample.

Table 1A-2-C. Residues of ETU (from M-45) in
East Coast Tomato Tailings

Sample	Amount Found ETU (ppm)	Average	* Corrected for Check	Average % Recovery	Corrected for Average % Recovery
Rep I check	<0.01; <0.01	<0.01			
Rep II check	<0.01; <0.01	<0.01			
Rep III check	<0.01; <0.01	<0.01			
Rep I 1x	0.01; <0.01	0.01	0.01	46.7	0.02
Rep II 1x	<0.01; <0.01	<0.01	<0.01		<0.01
Rep III 1x	<0.01; <0.01	<0.01	<0.01		<0.01
Rep I 4x	<0.01; <0.01	<0.01	<0.01	46.7	<0.01
Rep II 4x	<0.01; <0.01	<0.01	<0.01		<0.01
Rep III 4x	<0.01; 0.01				
	0.01; 0.03	0.01	0.01		0.02
	0.01; <0.01				

Recovery Studies ETU (ppm)

	Amount Added	Amount Found	* Corrected for Check	% Recovery	Average % Recovery
Rep I check	0.05 ETU	0.04	0.04	80	
Rep II check	0.05 ETU	0.01	0.01	20	
Rep III check	0.05 ETU	0.02	0.02	40	46.7

* Corrected by subtraction of amount found in check sample.

Table IA-2-D. Residues of ETU (from M-45) in
Canned Juice From East Coast Tomatoes

Sample	Amount Found ETU (ppm)	Average	* Corrected for Check	Average % Recovery	Corrected for Average % Recovery
Rep I check	<0.01, <0.01	<0.01			
Rep II check	0.01, 0.01	0.01			
Rep III check	0.01, <0.01	0.01			
Rep I 1x	0.06, 0.06	0.06	0.06	106.7	0.06
Rep II 1x	0.04, 0.04	0.04	0.03		0.03
Rep III 1x	0.02, 0.02	0.02	0.01		0.01
Rep I 4x	0.11, 0.10	0.11	0.11	106.7	0.10
Rep II 4x	0.11, 0.14	0.13	0.12		0.11
Rep III 4x	0.11, 0.08	0.10	0.09		0.08

Recovery Studies ETU (ppm)

	Amount Added	Amount Found	* Corrected For Check	% Recovery	Average % Recovery
Rep I check	0.05 ETU	0.05	0.05	100	
Rep II check	0.05 ETU	0.08	0.07	140	
Rep III check	0.05 ETU	0.05	0.04	80	106.7

* Corrected by subtraction of amount found in check sample.

Table IA-3-A. Residues of EBOC (from M-46) from
West Coast Unwashed Tomatoes

Sample	Amount Found EBOC (ppm)	Average	* Corrected for Check	Average % Recovery	Corrected for Avg. % Recovery
Rep I check	<0.1, <0.1	<0.1			
Rep II check	<0.1, <0.1	<0.1			
Rep III check	<0.1, <0.1	<0.1			
Rep I 1x	1.6, 1.4	1.5	1.5	96.2	1.6
Rep II 1x	1.8, 2.3	2.1	2.1		2.2
Rep III 1x	2.2, 2.1	2.2	2.2		2.3
Rep I 4x	5.4, 6.9	6.2	6.2	96.2	6.4
Rep II 4x	4.8, 5.8	5.3	5.3		5.6
Rep III 4x	5.1, 9.6	5.9	5.9		9.3

Recovery Studies EBOC (ppm)

	Amount Added	Amount Found	* Corrected for Check	% Recovery	Average % Recovery
Rep I check	1.2 EBOC**	1.2	1.2	100	
Rep II check	1.2 EBOC**	0.9	0.9	75	
Rep III check	1.2 EBOC**	1.3	1.3	108	
Rep I check	3.1 EBOC**	2.9	2.9	94	
Rep II check	3.1 EBOC**	2.8	2.8	90	
Rep III check	3.1 EBOC**	3.4	3.4	110	96.2

*Corrected by subtraction of amount found in check sample.

**Amount added has been corrected for % activity of 62.2.

Table IA-3-B. Residues of EBDC (from M-48) from
West Coast Washed Tomatoes

Sample	Amount Found EBDC (ppm)	Average	*Corrected for Check	Average % Recovery	Corrected for Avg. % Recovery
Rep I check	<0.1, <0.1	<0.1			
Rep II check	<0.1, <0.1	<0.1			
Rep III check	<0.1, <0.1	<0.1			
Rep I 1x	0.5, 0.4	0.5	0.5	88.7	0.6
Rep II 1x	0.4, 0.4	0.4	0.4		0.5
Rep III 1x	0.5, 0.5	0.5	0.5		0.6
Rep I 4x	0.8, 0.8	0.8	0.8	88.7	0.9
Rep II 4x	0.8, 0.8	0.8	0.8		0.9
Rep III 4x	0.9, 0.8	0.9	0.9		1.0

Recovery Studies EBDC (ppm)

	Amount Added	Amount Found	*Corrected for Check	% Recovery	Average % Recovery
Rep I check	1.2 EBDC**	1.0	1.0	83	
Rep II check	1.2 EBDC**	1.4	1.4	117	
Rep III check	1.2 EBDC**	0.7	0.7	58	
Rep I check	3.1 EBDC**	3.1	3.1	100	
Rep II check	3.1 EBDC**	3.3	3.3	106	
Rep III check	3.1 EBDC**	2.1	2.1	68	88.7

*Corrected by subtraction of amount found in check sample.

**Amount added has been corrected for % activity of 62.2.

Table 1A-3-C. Residues of EBDC (from M-45) from
West Coast Tomato Tailings

Sample	Amount Found EBDC (ppm)	Average	*Corrected for Check	Average % Recovery	Corrected for Avg. % Recovery
Rep I check	Values from East Coast used				
Rep II check					
Rep III check					
Rep I 1x	0.7, 0.4	0.6	0.6	94.7	0.6
Rep II 1x	0.4, 0.4	0.4	0.4		0.4
Rep III 1x	0.1, 0.1	0.1	0.1		0.1
Rep I 4x	0.7, 0.6	0.7	0.7	94.7	0.7
Rep II 4x	1.9, 1.4	1.6	1.6		1.6
Rep III 4x	1.9, 1.4	1.7	1.7		1.8

Recovery Studies EBDC (ppm)

	Amount Added	Amount Found	*Corrected for Check	% Recovery	Average % Recovery
Rep I check	Recovery studies from East Coast sample used due to lack of adequate sample.				
Rep II check					
Rep III check					
Rep I check					
Rep II check					
Rep III check					

*Check samples assumed to be <0.1 ppm.

**Average % Recovery from East Coast used.

Table 1A-3-D. Residues of EBDC (from M-45) from
Canned Juice from West Coast Tomatoes

Sample	Amount Found EBDC (ppm)	Average	* Corrected for Check	Average % Recovery	Corrected for Avg. % Recovery
Rep I check	<0.1, <0.1	<0.1			
Rep II check	<0.1, <0.1	<0.1			
Rep III check	<0.1, <0.1	<0.1			
Rep I 1x	0.4, 0.4	0.4	0.4	67.8	0.6
Rep II 1x	0.3, 0.3	0.3	0.3		0.4
Rep III 1x	0.4, 0.4	0.4	0.4		0.6
Rep I 4x	1.3, 1.3	1.3	1.3	67.8	1.9
Rep II 4x	0.8, 0.7	0.8	0.8		1.2
Rep III 4x	1.5, 1.6	1.6	1.6		2.4

Recovery Studies EBDC (ppm)

	Amount Added	Amount Found	* Corrected for Check	% Recovery	Average % Recovery
Rep I check	1.2 EBDC**	0.8	0.8	67	
Rep II check	1.2 EBDC**	0.8	0.8	67	
Rep III check	1.2 EBDC**	0.8	0.8	67	
Rep I check	3.1 EBDC**	1.8	1.8	58	
Rep II check	3.1 EBDC**	2.3	2.3	74	
Rep III check	3.1 EBDC**	2.3	2.3	74	67.8

*Corrected by subtraction of amount found in check sample.

**Amount added has been corrected for % activity of 62.2.

Table IA-4-A. Residues of ETU (from M-45) from
West Coast Unwashed Tomatoes

Sample	Amount Found ETU (ppm)	Average	*Corrected for Check	Average % Recovery	Corrected for Average % Recovery
Rep I check	0.01, <0.01	0.01			
Rep II check	<0.01, <0.01	<0.01			
Rep III check	<0.01, <0.01	<0.01			
Rep I 1x	0.01, <0.01	0.01	<0.01	86.7	<0.01
Rep II 1x	0.01, 0.01	0.01	0.01		0.01
Rep III 1x	0.01, <0.01	0.01	0.01		0.01
Rep I 4x	0.01, 0.01	0.01	<0.01	86.7	<0.01
Rep II 4x	0.02, 0.02	0.02	0.02		0.02
Rep III 4x	0.02, 0.05	0.04	0.04		0.05

Recovery Studies ETU (ppm)

	Amount Added	Amount Found	*Corrected for Check	% Recovery	Average % Recovery
Rep I check	0.05 ETU	0.03	0.03	60	
Rep II check	0.05 ETU	0.05	0.05	100	
Rep III check	0.05 ETU	0.05	0.05	100	86.7

*Corrected by subtraction of amount found in check sample.

Table 1A-4-B. Residues of ETU (from M-45) from
West Coast Washed Tomatoes

<u>Sample</u>	<u>Amount Found ETU (ppm)</u>	<u>Average</u>	<u>* Corrected for Check</u>	<u>Average Recovery</u>	<u>Corrected for Average Recovery</u>
Rep I check	<0.01, <0.01	<0.01			
Rep II check	0.01, <0.01	0.01			
Rep III check	<0.01, <0.01	<0.01			
Rep I 1x	<0.01, <0.01	<0.01	<0.01	60	<0.01
Rep II 1x	0.01, 0.01	0.01	<0.01		<0.01
Rep III 1x	0.01, <0.01	0.01	0.01		0.02
Rep I 4x	<0.01, 0.01	0.01	0.01	60	0.02
Rep II 4x	<0.01, <0.01	<0.01	<0.01		<0.01
Rep III 4x	<0.01, <0.01	<0.01	<0.01		<0.01

Recovery Studies ETU (ppm)

	<u>Amount Added</u>	<u>Amount Found</u>	<u>* Corrected for Check</u>	<u>Recovery</u>	<u>Average Recovery</u>
Rep I check	0.05 ETU	0.04	0.04	80	
Rep II check	0.05 ETU	0.01	0.01	20	
Rep III check	0.05 ETU	0.04	0.04	80	60

*Corrected by subtraction of amount found in check sample.

Table 1A-4-C. Residues of ETU (from 11-45) from
West Coast Tomato Tailings

Sample	Amount Found ETU (ppm)	Average	* Corrected for Check	Average % Recovery	Corrected for Average % Recovery
Rep I check	<0.01, <0.01	<0.01			
Rep II check	<0.01, <0.01	<0.01			
Rep III check	<0.01, <0.01	<0.01			
Rep I 1x	<0.01, <0.01	<0.01	<0.01	36.7	<0.01
Rep II 1x	<0.01, <0.01	<0.01	<0.01		<0.01
Rep III 1x	<0.01, <0.01	<0.01	<0.01		<0.01
Rep I 4x	0.02, 0.02	0.02	0.02	36.7	0.06
Rep II 4x	0.03, 0.03	0.03	0.03		0.08
Rep III 4x	<0.01, <0.01	<0.01	<0.01		<0.01

Recovery Studies ETU (ppm)

	Amount Added	Amount Found	* Corrected for Check	% Recovery	Average % Recovery
Rep I check	0.10 ETU	0.04	0.04	40	
Rep II check	0.10 ETU	0.05	0.05	50	
Rep III check	0.05 ETU	0.01	0.01	20	36.7

*Corrected by subtraction of amount in check sample.

Table 1A-4-D. Residues of ETU (from M-45) from
Canned Juice of West Coast Tomatoes

Sample	Amount Found ETU (ppm)	Average	*Corrected for Check	Average % Recovery	Corrected for Average % Recovery
Rep I check	0.02, 0.02	0.02			
Rep II check	0.01, 0.01	0.01			
Rep III check	0.01, 0.01	0.01			
Rep I 1x	0.15, 0.11	0.13	0.11	73.3	0.15
Rep II 1x	0.09, 0.09	0.09	0.08		0.11
Rep III 1x	0.07, 0.07	0.07	0.06		0.08
Rep I 4x	<0.01, 0.28	0.14	0.12	73.3	0.16
Rep II 4x	0.08, 0.13	0.11	0.10		0.14
Rep III 4x	0.08, 0.06	0.07	0.06		0.08

Recovery Studies ETU (ppm)

	Amount Added	Amount Found	*Corrected for Check	% Recovery	Average % Recovery
Rep I check	0.05 ETU	0.05	0.03	60	
Rep II check	0.05 ETU	0.07	0.06	120	
Rep III check	0.05 ETU	0.03	0.02	40	73.3

*Corrected by subtraction of amount found in check sample.

Table 1B-1-A. Residues of EBDC (from M-45) in
East Coast Unwashed Carrots

Sample	Amount Found EBDC (ppm)	Average	* Corrected for Check	Average % Recovery	Corrected for Average % Recovery
Rep I check	<0.1, <0.1	<0.1			
Rep II check	<0.1, <0.1	<0.1			
Rep III check	<0.1, <0.1	<0.1			
Rep I 1x	0.2, 0.3	0.3	0.3	82.0	0.4
Rep II 1x	0.5, 0.5	0.5	0.5		0.6
Rep III 1x	0.7, 0.7	0.7	0.7		0.9
Rep I 4x	0.4, 0.4	0.4	0.4	82.0	0.5
Rep II 4x	0.7, 0.7	0.7	0.7		0.9
Rep III 4x	0.9, 0.9	0.9	0.9		1.1

Recovery Studies EBDC (ppm)

	Amount Added	Amount Found	* Corrected for Check	% Recovery	Average % Recovery
Rep I check	1.2 EBDC**	0.8	0.8	67	
Rep II check	1.2 EBDC**	1.3	1.3	108	
Rep III check	1.2 EBDC**	0.9	0.9	75	
Rep I check	3.1 EBDC**	1.8	1.8	58	
Rep II check	3.1 EBDC**	3.1	3.1	100	
Rep III check	3.1 EBDC**	2.6	2.6	84	82.0

*Corrected by subtraction of amount found in check sample.

**Amount added has been corrected for % activity of 62.2.

Table IB-1-B. Residues of EBDC (from M-48) in
East Coast Washed Carrots

Sample	Amount Found EBDC (ppm)	Average	*Corrected for Check	Average % Recovery	Corrected for Average % Recovery
Rep I check	<0.1, <0.1	<0.1			
Rep II check	<0.1, <0.1	<0.1			
Rep III check	<0.1, <0.1	<0.1			
Rep I 1x	0.6, 0.6	0.6	0.5	70.6	0.7
Rep II 1x	0.1, <0.1	0.1	0.1		0.1
Rep III 1x	<0.1, <0.1	<0.1	<0.1		<0.1
Rep I 4x	0.8, 0.7	0.8	0.8	70.6	1.1
Rep II 4x	0.4, 0.4	0.4	0.4		0.6
Rep III 4x	0.3, 0.3	0.3	0.3		0.4

Recovery Studies EBDC (ppm)

	Amount Added	Amount Found	*Corrected for Check	% Recovery	Average % Recovery
Rep I check	1.2 EBDC**	1.0	1.0	83	
Rep II check	1.2 EBDC**	0.7	0.7	58	
Rep III check	1.2 EBDC**	0.9	0.9	75	
Rep I check	3.1 EBDC**	2.6	2.6	81	70.6
Rep II check	3.1 EBDC**	1.6	1.6	51	
Rep III check	3.1 EBDC**	2.1	2.1	68	

*Corrected by subtraction of amount found in check sample.

**Amount added has been corrected for % activity of 82.2.

Table IB-1-C. Residues of EBDC (from M-45) in
East Coast Blanched Diced Carrots

Sample	Amount Found EBDC (ppm)	Average	* Corrected for Check	Average % Recovery	Corrected for Average % Recovery
Rep I check	<0.1, <0.1	<0.1			
Rep II check	<0.1, <0.1	<0.1			
Rep III check	<0.1, <0.1	<0.1			
Rep I 1x	0.2, 0.1	0.2	0.2	87	0.2
Rep II 1x	0.1, 0.1	0.1	0.1		0.1
Rep III 1x	0.1, 0.1	0.1	0.1		0.1
Rep I 4x	0.4, 0.4	0.4	0.4	87	0.5
Rep II 4x	0.4, 0.4	0.4	0.4		0.5
Rep III 4x	0.3, 0.3	0.3	0.3		0.3

Recovery Studies EBDC (ppm)

	Amount Added	Amount Found	* Corrected for Check	% Recovery	Average % Recovery
Rep I check	1.2 EBDC**	0.8	0.8	67	
Rep II check	1.2 EBDC**	0.9	0.9	75	
Rep III check	1.2 EBDC**	1.5	1.5	125	
Rep I check	3.1 EBDC**	2.2	2.2	87	
Rep II check	3.1 EBDC**	2.6	2.6	84	
Rep III check	3.1 EBDC**	2.6	2.6	84	87

*Corrected by subtraction of amount found in check sample.

**Amount added has been corrected for % activity of 62.2.

Table IB-1-D. Residues of EBDC (from M-45) in
East Coast Frozen Diced Carrots

<u>Sample</u>	<u>Amount Found EBDC (ppm)</u>	<u>Average</u>	<u>* Corrected for Check</u>	<u>Average % Recovery</u>	<u>Corrected for Average % Recovery</u>
Rep I check	<0.1, <0.1	<0.1			
Rep II check	<0.1, <0.1	<0.1			
Rep III check	<0.1, <0.1	<0.1			
Rep I 1x	<0.1, <0.1	<0.1	<0.1	81.5	<0.1
Rep II 1x	<0.1, <0.1	<0.1	<0.1		<0.1
Rep III 1x	<0.1, <0.1	<0.1	<0.1		<0.1
Rep I 4x	<0.1, <0.1	<0.1	<0.1	81.5	<0.1
Rep II 4x	<0.1, <0.1	<0.1	<0.1		<0.1
Rep III 4x	<0.1, <0.1	<0.1	<0.1		<0.1

Recovery Studies EBDC (ppm)

	<u>Amount Added</u>	<u>Amount Found</u>	<u>* Corrected for Check</u>	<u>% Recovery</u>	<u>Average % Recovery</u>
Rep I check	1.2 EBDC**	0.8	0.8	67	
Rep II check	1.2 EBDC**	0.9	0.9	75	
Rep III check	1.2 EBDC**	1.1	1.1	92	
Rep I check	3.1 EBDC**	2.7	2.7	87	
Rep II check	3.1 EBDC**	2.6	2.6	84	
Rep III check	3.1 EBDC**	2.6	2.6	84	81.5

*Corrected by subtraction of amount found in check sample.

**Amount added has been corrected for % activity of 82.2.

Table IB-1-E. Residues of EBDC (from M-45) in
West Coast Canned Carrots

Sample	Amount found EBDC (ppm)	Average	* Corrected for Check	Average % Recovery	Corrected for Average % Recovery
Rep I check	<0.1, <0.1	<0.1			
Rep II check	<0.1, <0.1	<0.1			
Rep III check	<0.1, <0.1	<0.1			
Rep I 1x	0.1, 0.1	0.1	0.1	79.8	0.1
Rep II 1x	<0.1, <0.1	<0.1	<0.1		<0.1
Rep III 1x	<0.1, <0.1	<0.1	<0.1		<0.1
Rep I 4x	<0.1, <0.1	<0.1	<0.1	79.8	<0.1
Rep II 4x	<0.1, <0.1	<0.1	<0.1		<0.1
Rep III 4x	0.1, 0.1	0.1	0.1		0.1

Recovery Studies EBDC (ppm)

	Amount Added	Amount Found	* Corrected for Check	% Recovery	Average % Recovery
Rep I check	1.2 EBDC**	0.8	0.8	67	
Rep II check	1.2 EBDC**	0.8	0.8	67	
Rep III check	1.2 EBDC**	1.2	1.2	100	
Rep I check	3.1 EBDC**	2.4	2.4	77	
Rep II check	3.1 EBDC**	2.2	2.2	71	
Rep III check	3.1 EBDC**	3.1	3.0	97	79.8

*Corrected by subtraction of amount found in check sample.

**Amount added has been corrected for % activity of 62.2.

Table 1B-2-A. Residues of ETU (from M-46) in
East Coast Unwashed Carrots

<u>Sample</u>	<u>Amount Found ETU (ppm)</u>	<u>Average</u>	<u>*Corrected For Check</u>	<u>Average % Recovery</u>	<u>Corrected for Average % Recovery</u>
Rep I check	<0.01, <0.01	<0.01			
Rep II check	<0.01, <0.01	<0.01			
Rep III check	<0.01, <0.01	<0.01			
Rep I 1x	<0.01, <0.01	<0.01	<0.01	80	<0.01
Rep II 1x	<0.01, <0.01	<0.01	<0.01		<0.01
Rep III 1x	<0.01, <0.01	<0.01	<0.01		<0.01
Rep I 4x	0.01, 0.01	0.01	0.01	80	0.01
Rep II 4x	<0.01, <0.01	<0.01	<0.01		<0.01
Rep III 4x	0.01, <0.01	0.01	0.01		0.01

Recovery Studies ETU (ppm)

	<u>Amount Added</u>	<u>Amount Found</u>	<u>*Corrected For Check</u>	<u>% Recovery</u>	<u>Average % Recovery</u>
Rep I check	0.05 ETU	0.05	0.05	100	
Rep II check	0.05 ETU	0.04	0.04	80	
Rep III check	0.05 ETU	0.03	0.03	60	80

*Corrected by subtraction of amount found in check sample.

Table IB-2-B. Residues of ETU (from M-45) in
East Coast Washed Carrots

<u>Sample</u>	<u>Amount Found ETU (ppm)</u>	<u>Average</u>	<u>* Corrected for Check</u>	<u>Average Recovery</u>	<u>Corrected for Average Recovery</u>
Rep I check	<0.01, <0.01	<0.01			
Rep II check	<0.01, <0.01	<0.01			
Rep III check	<0.01, <0.01	<0.01			
Rep I 1x	0.01, <0.01	0.01	0.01	73.3	0.01
Rep II 1x	<0.01, <0.01	<0.01	<0.01		<0.01
Rep III 1x	<0.01, <0.01	<0.01	<0.01		<0.01
Rep I 4x	<0.01, 0.01	0.01	0.01	73.3	0.01
Rep II 4x	<0.01, <0.01	<0.01	<0.01		<0.01
Rep III 4x	<0.01, <0.01	<0.01	<0.01		<0.01

Recovery Studies ETU (ppm)

	<u>Amount Added</u>	<u>Amount Found</u>	<u>* Corrected for Check</u>	<u>Recovery</u>	<u>Average Recovery</u>
Rep I check	0.05 ETU	0.04	0.04	80	
Rep II check	0.05 ETU	0.03	0.03	60	
Rep III check	0.05 ETU	0.04	0.04	80	73.3

*Corrected by subtraction of amount found in check sample.

Table IB-2-C. Residues of ETU (from M-46) in
East Coast Blanched Diced Carrots

<u>Sample</u>	<u>Amount Found ETU (ppm)</u>	<u>Average</u>	<u>*Corrected for Check</u>	<u>Average % Recovery</u>	<u>Corrected for Average % Recovery</u>
Rep I check	<0.01, <0.01	<0.01			
Rep II check	<0.01, <0.01	<0.01			
Rep III check	<0.01, <0.01	<0.01			
Rep I 1x	<0.01, <0.01	<0.01	<0.01	73.3	<0.01
Rep II 1x	<0.01, <0.01	<0.01	<0.01		<0.01
Rep III 1x	<0.01, <0.01	<0.01	<0.01		<0.01
Rep I 4x	<0.01, <0.01	<0.01	<0.01	73.3	<0.01
Rep II 4x	<0.01, <0.01	<0.01	<0.01		<0.01
Rep III 4x	<0.01, <0.01	<0.01	<0.01		<0.01

Recovery Studies ETU (ppm)

	<u>Amount Added</u>	<u>Amount Found</u>	<u>*Corrected for Check</u>	<u>% Recovery</u>	<u>Average % Recovery</u>
Rep I check	0.05 ETU	0.03	0.03	60	
Rep II check	0.05 ETU	0.04	0.04	80	
Rep III check	0.05 ETU	0.04	0.04	80	73.3

*Corrected by subtraction of amount found in check sample.

Table 18-2-B. Residues of ETU (from M-45) in
East Coast Frozen Diced Carrots

<u>Sample</u>	<u>Amount Found ETU (ppm)</u>	<u>Average</u>	<u>* Corrected for Check</u>	<u>Average Recovery</u>	<u>Corrected for Average Recovery</u>
Rep I check	<0.01, <0.01	<0.01			
Rep II check	<0.01, <0.01	<0.01			
Rep III Check	<0.01, <0.01	<0.01			
Rep I 1x	<0.01, <0.01	<0.01	<0.01	80	<0.01
Rep II 1x	<0.01, <0.01	<0.01	<0.01		<0.01
Rep III 1x	<0.01, <0.01	<0.01	<0.01		<0.01
Rep I 4x	0.01, 0.01	0.01	0.01		0.01
Rep II 4x	0.01, 0.01	0.01	0.01		0.01
Rep III 4x	<0.01, <0.01	<0.01	<0.01		<0.01

Recovery Studies ETU (ppm)

	<u>Amount Added</u>	<u>Amount Found</u>	<u>* Corrected for Check</u>	<u>Average Recovery</u>	<u>Average Recovery</u>
Rep I check	0.05 ETU	0.04	0.04	80	
Rep II check	0.05 ETU	0.04	0.04	80	
Rep III check	0.04 ETU	0.04	0.04	80	80

*Corrected by subtraction of amount found in check sample.

Table IB-2-E. Residues of ETU (from M-45) in
East Coast Canned Carrots

<u>Sample</u>	<u>Amount Found ETU (ppm)</u>	<u>Average</u>	<u>* Corrected for Check</u>	<u>Average % Recovery</u>	<u>Corrected for Average % Recovery</u>
Rep I check	<0.01, <0.01	<0.01			
Rep II check	<0.01, <0.01	<0.01			
Rep III check	<0.01, <0.01	<0.01			
Rep I 1x	0.01, 0.01	0.01	0.01	40	0.03
Rep II 1x	0.01, 0.01	0.01	0.01		0.03
Rep III 1x	0.01, 0.01	0.01	0.01		0.03
Rep I 4x	0.02, 0.02	0.02	0.02	40	0.05
Rep II 4x	0.02, 0.02	0.02	0.02		0.05
Rep III 4x	0.02, 0.02	0.02	0.02		0.05

Recovery Studies ETU (ppm)

	<u>Amount Added</u>	<u>Amount Found</u>	<u>* Corrected for Check</u>	<u>% Recovery</u>	<u>Average % Recovery</u>
Rep I check	0.05 ETU	0.01	0.01	20	
Rep II check	0.05 ETU	0.02	0.02	40	
Rep III check	0.05 ETU	0.03	0.03	60	40

*Corrected by subtraction of amount found in check sample.

Table IB-3-A. Residues of EBDC (from M-45) in
West Coast Unwashed Carrots

<u>Sample</u>	<u>Amount Found EBDC (ppm)</u>	<u>Average</u>	<u>²Corrected for Check</u>	<u>Average % Recovery</u>	<u>Corrected for Average % Recovery</u>
Rep I check	<0.1, <0.1	<0.1			
Rep II check	<0.1, <0.1	<0.1			
Rep III check	0.1, 0.1	0.1			
Rep I 1x	0.2, 0.2	0.2	0.2	81.8	0.2
Rep II 1x	0.1, 0.1	0.1	0.1		0.1
Rep III 1x	0.1, 0.1	0.1	<0.1		<0.1
Rep I 4x	0.3, 0.3	0.3	0.3	81.8	0.4
Rep II 4x	0.4, 0.4	0.4	0.4		0.5
Rep III 4x	0.3, 0.4	0.4	0.3		0.4

Recovery Studies EBDC (ppm)

	<u>Amount Added</u>	<u>Amount Found</u>	<u>[*]Corrected for Check</u>	<u>% Recovery</u>	<u>Average % Recovery</u>
Rep I check	1.2 EBDC**	1.1	1.1	92	
Rep II check	1.2 EBDC**	1.0	1.0	83	
Rep III check	1.2 EBDC**	1.0	0.9	75	
Rep I check	2.9 EBDC**	1.9	1.9	61	
Rep II check	2.9 EBDC**	2.8	2.8	97	
Rep III check	2.9 EBDC**	2.5	2.4	83	81.8

*Corrected by subtraction of amount found in check sample.

**Amount added has been corrected for % activity of 57.7.

Table IB-3-B. Residues of EBDC (from M-45) in
West Coast Washed Carrots

Sample	Amount Found EBDC (ppm)	Average	*Corrected for Check	Average % Recovery	Corrected for Average % Recovery
Rep I check	<0.1, <0.1	<0.1			
Rep II check	0.1, 0.1	0.1			
Rep III check	<0.1, <0.1	<0.1			
Rep I 1x	<0.1, <0.1	<0.1	<0.1	79.3	<0.1
Rep II 1x	0.1, 0.1	0.1	<0.1		<0.1
Rep III 1x	0.2, 0.2	0.2	0.2		0.3
Rep I 4x	0.3, 0.2	0.3	0.3	79.3	0.4
Rep II 4x	0.3, 0.4	0.4	0.3		0.4
Rep III 4x	0.2, 0.2	0.2	0.2		0.3

Recovery Studies EBDC (ppm)

	Amount Added	Amount Found	*Corrected for Check	% Recovery	Average % Recovery
Rep I check	1.4 EBDC**	1.0	1.0	71	
Rep II check	1.4 EBDC**	1.0	0.9	64	
Rep III check	1.4 EBDC**	1.3	1.3	93	
Rep I check	3.4 EBDC**	2.4	2.4	71	
Rep II check	3.4 EBDC**	2.5	2.4	71	
Rep III check	3.4 EBDC**	3.6	3.6	106	79.3

*Corrected by subtraction of amount found in check sample.

**Amount added has been corrected for % activity of 67.6.

Table 1B-3-C. Residues of EBDC (From 2-45) in
West Coast Blanched Diced Carrots

Sample	Amount Found EBDC (ppm)	Average	*Corrected for Check	Average % Recovery	Corrected for Average % Recovery
Rep I check	<0.1, <0.1	<0.1			
Rep II check	<0.1, <0.1	<0.1			
Rep III check	0.1, 0.1	0.1			
Rep I 1x	0.1, 0.1	0.1	0.1	81.2	0.1
Rep II 1x	0.1, 0.1	0.1	0.1		0.1
Rep III 1x	0.1, 0.1	0.1	<0.1		<0.1
Rep I 4x	0.1, 0.1	0.1	0.1	81.2	0.1
Rep II 4x	0.1, 0.2	0.2	0.2		0.2
Rep III 4x	0.1, 0.1	0.1	<0.1		<0.1

Recovery Studies EBDC (ppm)

	Amount Added	Amount Found	*Corrected for Check	% Recovery	Average % Recovery
Rep I check	1.4 EBDC**	0.8	0.8	57	
Rep II check	1.4 EBDC**	1.1	1.1	79	
Rep III check	1.4 EBDC**	1.3	1.2	86	
Rep I check	3.4 EBDC**	2.4	2.4	71	
Rep II check	3.4 EBDC**	3.6	3.5	103	
Rep III check	3.4 EBDC**	3.2	3.1	91	81.2

*Corrected by subtraction of amount found in check sample.

**Amount added has been corrected for % activity of 67.6.

Table 15-3-D. Residues of EBDC (from M-45) in
West Coast Frozen Diced Carrots

<u>Sample</u>	<u>Amount Found EBDC (ppm)</u>	<u>Average</u>	<u>* Corrected for Check</u>	<u>Average % Recovery</u>	<u>Corrected for Average % Recovery</u>
Rep I check	<0.1, <0.1	<0.1			
Rep II check	<0.1, <0.1	<0.1			
Rep III check	<0.1, <0.1	<0.1			
Rep I 1x	<0.1, <0.1	<0.1	<0.1	87.8	<0.1
Rep II 1x	<0.1, <0.1	<0.1	<0.1		<0.1
Rep III 1x	0.1, 0.1	0.1	0.1		0.1
Rep I 4x	<0.1, <0.1	<0.1	<0.1	87.8	<0.1
Rep II 4x	0.1, 0.1	0.1	0.1		0.1
Rep III 4x	<0.1, <0.1	<0.1	<0.1		<0.1

Recovery Studies EBDC (ppm)

	<u>Amount Added</u>	<u>Amount Found</u>	<u>* Corrected for Check</u>	<u>% Recovery</u>	<u>Average % Recovery</u>
Rep I check	1.4 EBDC**	1.0	1.0	71	
Rep II check	1.4 EBDC**	1.1	1.1	79	
Rep III check	1.4 EBDC**	1.0	1.0	71	
Rep I check	3.4 EBDC**	3.3	3.3	97	
Rep II check	3.4 EBDC**	3.4	3.4	100	
Rep III check	3.4 EBDC**	3.7	3.7	109	87.8

*Corrected by subtraction of amount found in check sample.

**Amount added has been corrected for % activity of 87.6.

Table IB-3-E: Residues of EBOC (from M-45) in
West Coast Canned Carrots

Sample	Amount Found EBOC (ppm)	Average	*Corrected for Check	Average % Recovery	Corrected for Average % Recovery
Rep I check	<0.1, <0.1	<0.1			
Rep II check	<0.1, <0.1	<0.1			
Rep III check	<0.1, <0.1	<0.1			
Rep I 1x	<0.1, <0.1	<0.1	<0.1	82.8	<0.1
Rep II 1x	0.2, 0.2	0.2	0.2		0.2
Rep III 1x	0.2, 0.2	0.2	0.2		0.2
Rep I 4x	<0.1, <0.1	<0.1	<0.1	82.8	<0.1
Rep II 4x	0.2, 0.2	0.2	0.2		0.2
Rep III 4x	0.2, 0.2	0.2	0.2		0.2

Recovery Studies EBOC (ppm)

	Amount Added	Amount Found	*Corrected for Check	% Recovery	Average % Recovery
Rep I check	1.4 EBOC**	0.9	0.9	64	
Rep II check	1.4 EBOC**	0.9	0.9	64	
Rep III check	1.4 EBOC**	1.3	1.3	93	
Rep I check	3.4 EBOC**	3.0	3.0	88	
Rep II check	3.4 EBOC**	3.2	3.2	94	
Rep III check	3.4 EBOC**	3.3	3.2	94	82.8

*Corrected by subtraction of amount found in check sample.

**Amount added has been corrected for % activity of 87.6.

Table ID-4-A. Residues of ETU (from M-45) in
West Coast Unwashed Carrots

Sample	Amount Found ETU (ppm)	Average	*Corrected for Check	Average % Recovery	Corrected for Average % Recovery
Rep I check	<0.01, <0.01	<0.01			
Rep II check	<0.01, <0.01	<0.01			
Rep III check	<0.01, <0.01	<0.01			
Rep I 1x	0.01, 0.01	0.01	0.01	60	0.02
Rep II 1x	<0.01, <0.01	<0.01	<0.01		<0.01
Rep III 1x	<0.01, <0.01	<0.01	<0.01		<0.01
Rep I 4x	<0.01, <0.01	<0.01	<0.01	60	<0.01
Rep II 4x	<0.01, <0.01	<0.01	<0.01		<0.01
Rep III 4x	<0.01, <0.01	<0.01	<0.01		<0.01

Recovery Studies ETU (ppm)

	Amount Added	Amount Found	*Corrected for Check	% Recovery	Average % Recovery
Rep I check	0.05 ETU	0.03	0.03	60	
Rep II check	0.05 ETU	0.04	0.04	80	
Rep III check	0.05 ETU	0.02	0.02	40	60

*Corrected by subtraction of amount found in check sample.

Table IB-4-B, Residues of ETU (from M-46) in
West Coast Washed Carrots

Sample	Amount Found ETU (ppm)	Average	* Corrected for Check	Average % Recovery	Corrected for Average % Recovery
Rep I check	<0.01, <0.01	<0.01			
Rep II check	<0.01, <0.01	<0.01			
Rep III check	<0.01, <0.01	<0.01			
Rep I 1x	0.01, <0.01	0.01	0.01	66.7	0.02
Rep II 1x	<0.01, <0.01	<0.01	<0.01		<0.01
Rep III 1x	<0.01, <0.01	<0.01	<0.01		<0.01
Rep I 4x	<0.01, <0.01	<0.01	<0.01	66.7	<0.01
Rep II 4x	<0.01, <0.01	<0.01	<0.01		<0.01
Rep III 4x	<0.01, <0.01	<0.01	<0.01		<0.01

Recovery Studies ETU (ppm)

	Amount Added	Amount Found	* Corrected for Check	% Recovery	Average % Recovery
Rep I check	0.05 ETU	0.03	0.03	60	
Rep II check	0.05 ETU	0.03	0.03	60	
Rep III check	0.05 ETU	0.04	0.04	80	66.7

*Corrected by subtraction of amount found in check sample.

Table IB-4-C. Residues of ETU (from M-45) in
West Coast Blanched Diced Carrots

Sample	Amount Found ETU (ppm)	Average	* Corrected for Check	Average % Recovery	Corrected for Average % Recovery
Rep I check	<0.01, <0.01	<0.01			
Rep II check	<0.01, <0.01	<0.01			
Rep III check	<0.01, <0.01	<0.01			
Rep I 1x	<0.01, <0.01	<0.01	<0.01	53.3	<0.01
Rep II 1x	<0.01, <0.01	<0.01	<0.01		<0.01
Rep III 1x	<0.01, <0.01	<0.01	<0.01		<0.01
Rep I 4x	<0.01, <0.01	<0.01	<0.01	53.3	<0.01
Rep II 4x	<0.01, <0.01	<0.01	<0.01		<0.01
Rep III 4x	<0.01, <0.01	<0.01	<0.01		<0.01

Recovery Studies ETU (ppm)

	Amount Added	Amount Found	* Corrected for Check	% Recovery	Average % Recovery
Rep I check	0.05 ETU	0.04	0.04	80	
Rep II check	0.05 ETU	0.02	0.02	40	
Rep III check	0.05 ETU	0.02	0.02	40	53.3

*Corrected by subtraction of amount found in check sample.

Table IB-4-D. Residues of ETU (from M-45) in
West Coast Frozen Diced Carrots

<u>Sample</u>	<u>Amount Found ETU (ppm)</u>	<u>Average</u>	<u>* Corrected for Check</u>	<u>Average % Recovery</u>	<u>Corrected for Average % Recovery</u>
Rep I check	<0.01, <0.01	<0.01			
Rep II check	<0.01, <0.01	<0.01			
Rep III check	<0.01, <0.01	<0.01			
Rep I 1x	<0.01, <0.01	<0.01	<0.01	93.3	<0.01
Rep II 1x	<0.01, <0.01	<0.01	<0.01		<0.01
Rep III 1x	<0.01, <0.01	<0.01	<0.01		<0.01
Rep I 4x	0.01, <0.01	0.01	0.01	93.3	0.01
Rep II 4x	<0.01, <0.01	<0.01	<0.01		<0.01
Rep III 4x	<0.01, <0.01	<0.01	<0.01		<0.01

Recovery Studies ETU (ppm)

	<u>Amount Added</u>	<u>Amount Found</u>	<u>* Corrected for Check</u>	<u>% Recovery</u>	<u>Average % Recovery</u>
Rep I check	0.05 ETU	0.05	0.05	100	
Rep II check	0.05 ETU	0.05	0.05	100	
Rep III check	0.05 ETU	0.04	0.04	80	93.3

*Corrected by subtraction of amount found in check sample.

Table IB-4-E. Residues of ETU (from M-45) in
West Coast Canned Carrots

Sample	Amount Found ETU (ppm)	Average	* Corrected for Check	Average % Recovery	Corrected for Average % Recovery
Rep I check	<0.01, <0.01	<0.01			
Rep II check	<0.01, <0.01	<0.01			
Rep III check	<0.01, <0.01	<0.01			
Rep I 1x	<0.01, <0.01	<0.01	<0.01	86.7	<0.01
Rep II 1x	<0.01, <0.01	<0.01	<0.01		<0.01
Rep III 1x	<0.01, 0.01	0.01	0.01		0.01
Rep I 4x	0.01, <0.01	0.01	0.01	86.7	0.01
Rep II 4x	0.01, 0.01	0.01	0.01		0.01
Rep III 4x	0.01, 0.01	0.01	0.01		0.01

Recovery Studies ETU (ppm)

	Amount Added	Amount Found	* Corrected for Check	% Recovery	Average % Recovery
Rep I check	0.05 ETU	0.04	0.04	80	
Rep II check	0.05 ETU	0.05	0.05	100	
Rep III check	0.05 ETU	0.04	0.04	80	86.7

*Corrected by subtraction of amount found in check sample.

Table 10-1-A. Residues of EBOC (from M-45) in
East Coast Fresh Unwashed Spinach

Sample	Amount Found EBOC (ppm)	Average	*Corrected for Check	Average % Recovery	Corrected for Average % Recovery
Rep I check	0.1, 0.2, 0.2	0.2			
Rep II check	0.2, 0.3, 0.2	0.2			
Rep III check	0.1, 0.1, 0.1, 0.2	0.1			
Rep I 1x	1.1, 0.9, 2.1	1.4	1.2	68.2	1.8
Rep II 1x	1.9, 1.9, 2.0, 1.3	1.8	1.6		2.3
Rep III 1x	2.4, 2.3, 2.5	2.4	2.3		3.4
Rep I 4x	3.2, 3.2	3.2	3.0	68.2	4.4
Rep II 4x	5.6, 5.0, 4.8	5.1	4.9		7.2
Rep III 4x	5.3, 6.1	5.7	5.6		8.2

Recovery Studies EBOC (ppm)

	Amount Added	Amount Found	*Corrected for Check	% Recovery	Average % Recovery
Rep I check	1.5 EBOC**	0.6, 1.0, 1.4, 1.0	0.6, 0.8, 1.7, 0.8	56	
Rep II check	1.5 EBOC**	1.0, 1.6, 0.9	0.8, 1.3, 0.7	62	
Rep III check	1.5 EBOC**	1.4, 1.0, 1.1, 1.2	1.2, 0.8, 0.9, 1.0	65	
Rep I check	3.7 EBOC**	3.3, 3.1	3.1, 2.9	81	
Rep II check	3.7 EBOC**	3.5, 2.8	3.3, 2.6	80	
Rep III check	3.7 EBOC**	2.8, 3.7	2.6, 3.5	83	68.2

*Corrected by subtraction of amount found in check sample.

**Amount added has been corrected for % activity of 74.7.

Table 10-1-B. Residues of EBDC (from M-45) in
East Coast Fresh Washed Spinach

Sample	Amount Found EBDC (ppm)	Average	*Corrected for Check	Average % Recovery	Corrected for Average % Recovery
Rep I check	0.1, 0.1, 0.1, 0.2	0.1			
Rep II check	<0.1, 0.1, 0.2	0.1			
Rep III check	<0.1, <0.1	<0.1			
Rep I 1x	1.0, 1.1, 1.1	1.1	1.0	84.5	1.2
Rep II 1x	0.9, 1.4	1.2	1.1		1.3
Rep III 1x	1.2, 2.6, 1.1	1.6	1.6		1.9
Rep I 4x	2.2, 2.1	2.2	2.1	84.5	2.6
Rep II 4x	2.4, 2.2, 2.1	2.2	2.1		2.6
Rep III 4x	2.6, 2.6, 2.2	2.5	2.5		3.0

Recovery Studies EBDC (ppm)

	Amount Added	Amount Found	*Corrected for Check	% Recovery	Average % Recovery
Rep I check	1.5 EBDC**	1.0, 1.2, 1.5	0.9, 1.1, 1.4	76	
Rep II check	1.5 EBDC**	1.8, 1.6	1.7, 1.5	107	
Rep III check	1.5 EBDC**	1.0, 1.0, 1.6	1.0, 1.0, 1.6	80	
Rep I check	3.7 EBDC**	3.2, 3.6	3.3, 3.7	95	
Rep II check	3.7 EBDC**	3.9, 3.4	3.8, 3.3	96	
Rep III check	3.7 EBDC**	3.1, 2.9, 1.7	3.1, 2.9, 1.7	69	84.5

*Corrected by subtraction of amount found in check sample.

**Amount added has been corrected for activity of 74.7.

Table IC-1-C. Residues of EBDC (from M-45) in
East Coast Frozen Blanched Spinach

Sample	Amount Found EBDC (ppm)	Average	*Corrected for Check	Average % Recovery	Corrected for Average % Recovery
Rep I check	<0.1, <0.1, <0.1, <0.1, <0.1, 0.3 ¹ , 0.4 ¹	<0.1			
Rep II check	<0.1, <0.1, 0.7 ¹ , <0.1	<0.1			
Rep III check	0.1, 0.2, 0.1, 0.1, 0.1, 0.2	0.1			
Rep I 1x	<0.1, 0.2, 0.2	0.1	0.1	88.4	0.1
Rep II 1x	0.2, 0.1	0.2	0.2		0.2
Rep III 1x	0.3, 0.2, 0.2	0.2	0.1		0.1
Rep I 4x	0.3, 0.3	0.3	0.3	88.4	0.4
Rep II 4x	0.5, 0.5	0.5	0.5		0.6
Rep III 4x	0.2, 0.4	0.3	0.2		0.2

Recovery Studies EBDC (ppm)

	Amount Added	Amount Found	*Corrected for Check	% Recovery	Average % Recovery
Rep I check	1.5 EBDC**	0.9, 1.4, 1.5, 1.5	0.9, 1.4, 1.5, 1.6	90	
Rep II check	1.5 EBDC**	1.2, 1.2, 1.6	1.2, 1.2, 1.6	89	
Rep III check	1.5 EBDC**	1.1, 1.1, 1.4, 1.3, 1.9, 1.7	1.0, 1.0, 1.3, 1.2, 1.8, 1.6	58	
Rep I check	3.7 EBDC**	4.3, 3.1	4.3, 3.1	100	
Rep II check	3.7 EBDC**	3.9, 3.3, 3.8, 2.9, 3.7	3.9, 3.3, 3.8, 2.9, 3.7	95	
Rep III check	3.7 EBDC**	2.4	2.3	6%	88.4

*Corrected by subtraction of amount found in check sample.

**Amount added has been corrected for % activity of 74.7.

¹Results excluded from calculation of average due to contamination.

Table IC-1-D. Residues of EBOC (from M-45) in
East Coast Canned Spinach

Sample	Amount Found EBOC (ppm)	Average	* Corrected for Check	Average % Recovery	Corrected for Average % Recovery
Rep I check	0.1, 0.1, 0.1	0.1			
Rep II check	0.1, 0.1	0.1			
Rep III check	0.2, 0.2	0.2			
Rep I 1x	0.1, 0.1	0.1	<0.1	89.7	<0.1
Rep II 1x	0.1, 0.1	0.1	<0.1		<0.1
Rep III 1x	<0.1, <0.1	<0.1	<0.1		<0.1
Rep I 4x	0.1, 0.1, 0.1	0.1	<0.1	89.7	<0.1
Rep II 4x	0.2, 0.2	0.2	0.1		0.1
Rep III 4x	<0.1, 0.1, 0.1	0.1	<0.1		<0.1

Recovery Studies EBOC (ppm)

	Amount Added	Amount Found	* Corrected for Check	% Recovery	Average % Recovery
Rep I check	1.5 EBOC**	1.2, 1.4, 1.8,	1.6, 1.4, 1.8,	99	
		1.2, 1.4	1.2, 1.4		
Rep II check	1.5 EBOC**	1.2, 1.4	1.1, 1.3	80	
Rep III check	1.5 EBOC**	1.3, 1.3, 1.3,	1.1, 1.1, 1.1,	77	
		1.5	1.3		
Rep I check	3.7 EBOC**	3.8, 4.0, 3.8	3.7, 3.9, 3.7	99	
Rep II check	3.7 EBOC**	2.8, 3.7, 3.9,	2.7, 3.6, 3.8,	94	
		3.4	3.3		
Rep III check	3.7 EBOC**	3.1	2.9	78	89.7

*Corrected by subtraction of amount found in check sample.

**Amount added has been corrected for % activity of 96.4%.

Table IC-2-A. Residues of ETU (from M-45) in
East Coast Unwashed Fresh Spinach

Sample	Amount Found ETU (ppm)	Average	*Corrected for Check	Average % Recovery	Corrected for Average % Recovery
Rep I check	<0.01, <0.01	<0.01			
Rep II check	<0.01, <0.01	<0.01			
Rep III check	<0.01, <0.01	<0.01			
Rep I 1x	<0.01, <0.01	<0.01	<0.01	73.3	<0.01
Rep II 1x	<0.01, <0.01	<0.01	<0.01		<0.01
Rep III 1x	<0.01, 0.01	0.01	0.01		0.01
Rep I 4x	0.01, 0.01	0.01	0.01	73.3	0.01
Rep II 4x	0.01, 0.01	0.01	0.01		0.01
Rep III 4x	0.01, 0.01	0.01	0.01		0.01

Recovery Studies ETU (ppm)

	Amount Added	Amount Found	*Corrected for Check	% Recovery	Average % Recovery
Rep I check	0.05 ETU	0.03	0.03	60	
Rep II check	0.05 ETU	0.03	0.03	60	
Rep III check	0.05 ETU	0.04	0.04	80	73.3

*Corrected by subtraction of amount found in check sample.

Table IC-2-B. Residues of ETU (from M-45) in
East Coast Vashed Fresh Spinach

Sample	Amount Found ETU (ppm)	Average	*Corrected for Check	Average % Recovery	Corrected for Average % Recovery
Rep I check	<0.01, <0.01	<0.01			
Rep II check	<0.01, <0.01	<0.01			
Rep III check	<0.01, <0.01	<0.01			
Rep I 1x	<0.01, <0.01	<0.01	<0.01	86	<0.01
Rep II 1x	<0.01, <0.01	<0.01	<0.01		<0.01
Rep III 1x	<0.01, <0.01	<0.01	<0.01		<0.01
Rep I 4x	0.01, 0.01	0.01	0.01	80	0.01
Rep II 4x	<0.01, 0.01	0.01	0.01		0.01
Rep III 4x	0.01, 0.01	0.01	0.01		0.01

Recovery Studies ETU (ppm)

	Amount Added	Amount Found	*Corrected for Check	% Recovery	Average % Recovery
Rep I check	0.05 ETU	0.04	0.04	80	
Rep II check	0.05 ETU	0.04	0.04	80	
Rep III check	0.05 ETU	0.07	0.07	140	80

*Corrected by subtraction of amount found in check sample.

Table IC-2-C. Residues of ETU (from M-45) in
East Coast Frozen Blanched Spinach

Sample	Amount Found ETU (ppm)	Average	*Corrected for Check	Average % Recovery	Corrected for Average % Recovery
Rep I check	<0.01, <0.01	<0.01			
Rep II check	<0.01, <0.01	<0.01			
Rep III check	<0.01, <0.01	<0.01			
Rep I 1x	0.02, 0.02	0.02	0.02	73.3	0.03
Rep II 1x	0.03, 0.02	0.03	0.03		0.04
Rep III 1x	0.03, 0.03	0.03	0.03		0.04
Rep I 4x	0.02, 0.03	0.03	0.03	73.3	0.04
Rep II 4x	0.04, 0.06	0.05	0.05		0.07
Rep III 4x	0.08, 0.08	0.08	0.08		0.11

Recovery Studies ETU (ppm)

	Amount Added	Amount Found	*Corrected for Check	% Recovery	Average % Recovery
Rep I check	0.05 ETU	0.03	0.03	60	
Rep II check	0.05 ETU	0.04	0.04	80	
Rep III check	0.05 ETU	0.04	0.04	80	73.3

*Corrected by subtraction of amount found in check sample.

Table IC-2-0. Residues of ETU (from M-46) in
East Coast Canned Spinach

Sample	Amount Found ETU (ppm)	Average	*Corrected for Check	Average % Recovery	Corrected for Average % Recovery
Rep I check	0.01, 0.01	0.01			
Rep II check	0.02, 0.01	0.02			
Rep III check	0.01, 0.01	0.01			
Rep I 1x	0.11, 0.12	0.12	0.11	66.7	0.16
Rep II 1x	0.14, 0.14	0.14	0.12		0.18
Rep III 1x	0.14, 0.14	0.14	0.13		0.19
Rep I 4x	0.30, 0.26	0.28	0.27	66.7	0.40
Rep II 4x	0.31, 0.30	0.35	0.33		0.49
Rep III 4x	0.28, 0.30	0.29	0.28		0.42

Recovery Studies ETU (ppm)

	Amount Added	Amount Found	*Corrected for Check	% Recovery	Average % Recovery
Rep I check	0.06 ETU	0.06	0.04	80	
Rep II check	0.06 ETU	0.06	0.03	80	
Rep III check	0.06 ETU	0.04	0.03	80	66.7

*Corrected by subtraction of amount found in check sample.

Table IC-3-A. Residues of EBDC (from M-45) in
West Coast Unwashed Spinach

Sample	Amount Found EBDC (ppm)		Average	* Corrected for Check	Average % Recovery	Corrected for Average % Recovery
Rep I check	<0.1,	<0.1	<0.1			
Rep II check	0.3,	0.3	0.3			
Rep III check	0.4,	0.6	0.5			
Rep I 1x	23.7,	23.6	23.6	23.6	87	27.1
Rep II 1x	42.9,	41.6	42.2	41.9		48.2
Rep III 1x	122.5,	70.7	96.6	96.1		110.5
Rep I 4x	204.4,	172.3	188.6	188.6	87	216.8
Rep II 4x	201.3,	180.0	190.7	190.4		218.9
Rep III 4x	180.6,	206.9	193.8	193.3		222.1

Recovery Studies EBDC (ppm)

	Amount Added	Amount Found	* Corrected for Check	% Recovery	Average % Recovery
Rep I check	1.0 EBDC**	0.6	0.6	60	
Rep II check	1.0 EBDC**	1.0	0.7	70	
Rep III check	1.0 EBDC**	1.5	1.0	100	
Rep I check	2.5 EBDC**	2.0	2.0	80	
Rep II check	2.5 EBDC**	2.4	2.1	84	
Rep III check	2.5 EBDC**	3.7	3.2	128	87

*Corrected by subtraction of amount found in check sample.

**Amount added has been corrected for % activity or 49.8.

Table IC-3-B. Residues of EBDC (from H-45) in
West Coast Washed Spinach

Sample	Amount Found EBDC (ppm)	Average	* Corrected for Check	Average % Recovery	Corrected for Average % Recovery
Rep I check	0.1, 0.1	0.1			
Rep II check	0.1, 0.1	0.1			
Rep III check	0.4, 0.4	0.4			
Rep I 1x	8.6, 6.9	7.8	7.7	66.7	11.5
Rep II 1x	8.6, 6.9	8.6	8.6		0.2
Rep III 1x	6.6, 6.7	6.7	6.3		9.4
Rep I 4x	38.5, 44.1	41.3	41.2	66.7	61.8
Rep II 4x	43.8, 45.3	44.6	44.5		66.7
Rep III 4x	30.0, 34.3	32.2	31.8		47.7

Recovery Studies EBDC (ppm)

	Amount Added	Amount Found	* Corrected for Check	% Recovery	Average % Recovery
Rep I check	1.0 EBDC**	0.8	0.7	70	
Rep II check	1.0 EBDC**	0.9	0.8	80	
Rep III check	1.0 EBDC**	0.9	0.5	50	
Rep I check	2.5 EBDC**	2.0	1.9	76	
Rep II check	2.5 EBDC**	1.6	1.5	60	
Rep III check	2.5 EBDC**	2.0	1.6	64	66.7

*Corrected by subtraction of amount found in check sample.

**Amount added has been corrected for % activity of 49.8.

Table IC-3-C. Residues of EBDC (from M-45) in
West Coast Frozen Spinach

<u>Sample</u>	<u>Amount Found EBDC (ppm)</u>	<u>Average</u>	<u>*Corrected for Check</u>	<u>Average % Recovery</u>	<u>Corrected for Average % Recovery</u>
Rep I check	0.2, 0.2	0.2			
Rep II check	0.1, 0.1	0.1			
Rep III check	0.1, 0.1	0.1			
Rep I 1x	0.2, 0.3	0.3	0.1	79.3	0.1
Rep II 1x	0.2, 0.2	0.2	0.6		0.8
Rep III 1x	0.6, 0.8	0.7	0.6		0.3
Rep I 4x	1.4, 1.4	1.4	1.2	79.3	1.6
Rep II 4x	2.0, 2.0	2.0	1.9		2.4
Rep III 4x	1.7, 1.6	1.7	1.6		2.0

Recovery Studies EBDC (ppm)

	<u>Amount Added</u>	<u>Amount Found</u>	<u>*Corrected for Check</u>	<u>% Recovery</u>	<u>Average % Recovery</u>
Rep I check	1.0 EBDC**	0.7	0.6	60	
Rep II check	1.0 EBDC**	0.9	0.8	80	
Rep III check	1.0 EBDC**	1.0	0.9	90	
Rep I check	2.5 EBDC**	2.1	1.9	76	
Rep II check	2.5 EBDC**	2.0	1.9	76	
Rep III check	2.5 EBDC**	2.7	2.6	104	79.3

*Corrected by subtraction of amount found in check sample.

**Amount added has been corrected for % activity of 49.8.

Table IC-3-D: Residues of EBDC (from M-45) in
West Coast Canned Spinach

Sample	Amount Found EBDC (ppm)	Average	*Corrected for Check	Average % Recovery	Corrected for Average % Recovery
Rep I check	<0.1, <0.1	<0.1			
Rep II check	0.1, 0.1	0.1			
Rep III check	0.1, 0.1	0.1			
Rep I 1x	<0.1, 0.1	0.1	0.1	76.0	0.1
Rep II 1x	0.1, 0.1	0.1	<0.1		<0.1
Rep III 1x	0.1, 0.2	0.2	0.1		0.1
Rep I 4x	0.2, 0.1	0.2	0.2	76.0	0.2
Rep II 4x	0.1, 0.1	0.1	<0.1		<0.1
Rep III 4x	0.1, 0.1	0.1	<0.1		<0.1

Recovery Studies EBDC (ppm)

	Amount Added	Amount Found	*Corrected for Check	% Recovery	Average % Recovery
Rep I check	1.0 EBDC**	0.9	0.9	90	
Rep II check	1.0 EBDC**	0.8	0.7	70	
Rep III check	1.0 EBDC**	0.5	0.4	40	
Rep I check	2.5 EBDC**	2.3	2.3	92	
Rep II check	2.5 EBDC**	2.0	1.8	72	
Rep III check	2.5 EBDC**	2.4	2.3	92	76.0

*Corrected by subtraction of amount found in check sample.

**Amount added has been corrected for % activity of 49.8.

Table IC-4-A. Residues of ETU (from M-45) in
West Coast Unwashed Spinach

<u>Sample</u>	<u>Amount Found ETU (ppm)</u>	<u>Average</u>	<u>*Corrected for Check</u>	<u>Average % Recovery</u>	<u>Corrected for Average % Recovery</u>
Rep I check	<0.01, <0.01	<0.01			
Rep II check	0.02, 0.01	0.02			
Rep III check	0.01, 0.01	0.01			
Rep I 1x	0.09, 0.11	0.10	0.10	46.7	0.21
Rep II 1x	0.20, 0.14	0.17	0.16		0.32
Rep III 1x	0.19, 0.28	0.24	0.23		0.49
Rep I 4x	0.46, 0.62	0.56	0.56	46.7	1.16
Rep II 4x	0.43, 0.43	0.43	0.41		0.88
Rep III 4x	0.89, 1.16	1.03	1.02		2.16

Recovery Studies ETU (ppm)

	<u>Amount Added</u>	<u>Amount Found</u>	<u>*Corrected for Check</u>	<u>% Recovery</u>	<u>Average % Recovery</u>
Rep I check	0.05 ETU	0.03	0.03	60	
Rep II check	0.05 ETU	0.03	0.01	20	
Rep III check	0.05 ETU	0.04	0.03	60	46.7

*Corrected by subtraction of amount found in check sample.

Table IC-4-B. Residues of ETU (from M-45) in
West Coast Washed Spinach

Sample	Amount Found ETU (ppm)	Average	* Corrected for Check	Average % Recovery	Corrected for Average % Recovery
Rep I check	<0.01, <0.01	<0.01			
Rep II check	<0.01, <0.01	<0.01			
Rep III check	<0.01, <0.01	<0.01			
Rep I 1x	0.01, 0.01	0.01	0.01	66.7	0.01
Rep II 1x	0.01, 0.01	0.01	0.01		0.01
Rep III 1x	0.02, 0.02	0.02	0.02		0.03
Rep I 4x	0.05, 0.05	0.05	0.05	66.7	0.07
Rep II 4x	0.04, 0.03	0.04	0.04		0.06
Rep III 4x	0.07, 0.08	0.08	0.08		0.12

Recovery Studies ETU (ppm)

	Amount Added	Amount Found	* Corrected for Check	% Recovery	Average % Recovery
Rep I check	0.05 ETU	0.04	0.04	80	
Rep II check	0.05 ETU	0.03	0.03	60	
Rep III check	0.05 ETU	0.03	0.03	60	66.7

*Corrected by subtraction of amount found in check sample.

Table IC-4-C. Residues of ETU (from M-45) in
West Coast Frozen Spinach

<u>Sample</u>	<u>Amount Found ETU (ppm)</u>	<u>Average</u>	<u>* Corrected for Check</u>	<u>Average % Recovery</u>	<u>Corrected for Average % Recovery</u>
Rep I check	0.01; 0.01	0.01			
Rep II check	<0.01; <0.01	<0.01			
Rep III check	0.01; 0.01	0.01			
Rep I 1x	0.13; 0.17	0.15	0.14	53.3	0.26
Rep II 1x	0.39; 0.27	0.33	0.33		0.62
Rep III 1x	0.31; 0.36	0.34	0.33		0.62
Rep I 4x	0.33; 0.46	0.39	0.38	53.3	0.71
Rep II 4x	2.84; 3.28	3.06	3.06		5.74
Rep III 4x	2.67; 2.42	2.55	2.54		4.78

Recovery Studies ETU (ppm)

	<u>Amount Added</u>	<u>Amount Found</u>	<u>* Corrected for Check</u>	<u>% Recovery</u>	<u>Average % Recovery</u>
Rep I check	0.05 ETU	0.02	0.01	20	
Rep II check	0.05 ETU	0.04	0.04	80	
Rep III check	0.05 ETU	0.04	0.03	60	53.3

*Corrected by subtraction of amount found in check sample.

Table IC-4-D. Residues of ETU (from M-45) in
West Coast Canned Spinach

Sample	Amount Found ETU (ppm)	Average	* Corrected for Check	Average % Recovery	Corrected for Average % Recovery
Rep I check	<0.01, <0.01	<0.01			
Rep II check	<0.01, <0.01	<0.01			
Rep III check	0.02, 0.02	0.02			
Rep I 1x	0.67, 0.60	0.74	0.74	106.7	0.69
Rep II 1x	0.64, 0.51	0.53	0.53		3.50
Rep III 1x	0.97, 1.06	1.02	1.00		0.94
Rep I 4x	2.36, 2.31	2.33	2.33	106.7	2.18
Rep II 4x	2.71, 3.84	3.28	3.28		3.07
Rep III 4x	2.67, 3.16	2.92	2.90		2.72

Recovery Studies ETU (ppm)

	Amount Added	Amount Found	* Corrected for Check	% Recovery	Average % Recovery
Rep I check	0.06 ETU	0.06	0.06	100	
Rep II check	0.06 ETU	0.06	0.06	100	
Rep III check	0.06 ETU	0.08	0.06	120	106.7

*Corrected by subtraction of amount found in check sample.

APPENDIX C

INDIVIDUAL DATA FROM SUMMARY TABLE 4

Residues of EBOC and ETU (from Dithane M-22[®])
in Tomatoes and Spinach at Process Stages

Table IIA-1-A. Residues of EBDC (from M-22) from
West Coast Unwashed Tomatoes

Sample	Amount Found EBDC (ppm)		Average	*Corrected for Check	Avg. % Recovery	Corrected for Avg. % Recovery
Rep I Check	0.2	0.2	0.2			
Rep II Check	0.1	0.1	0.1			
Rep III Check	0.2	0.2	0.2			
Rep I 1x	2.0	2.3	2.2	2.0	77.8	2.6
Rep II 1x	1.8	1.6	1.7	1.6		2.1
Rep III 1x	2.1	2.1	2.1	1.9		2.4
Rep I 4x	7.5	7.9	7.7	7.5	77.8	9.6
Rep II 4x	4.3	4.0	4.0	4.7		6.0
Rep III 4x	6.0	6.6	6.7	6.5		8.4

Recovery Studies EBDC (ppm)

	Amount Added	Amount Found	*Corrected for Check	% Recovery	Avg. % Recovery
Rep I Check	1.2 EBDC**	1.2	1.0	83	
Rep II Check	1.2 EBDC**	1.1	1.0	83	
Rep III Check	1.2 EBDC**	1.1	0.9	75	
Rep I Check	3.0 EBDC**	2.6	2.4	80	
Rep II Check	3.0 EBDC**	2.3	2.2	73	
Rep III Check	3.0 EBDC**	2.5	2.3	77	77.8

*Corrected by subtraction of amount found in check sample.

**Amount added has been corrected for % activity of 60.

Table IIA-1-B. Residues of EBDC (from M-22) from
West Coast Washed Tomatoes

Sample	Amount Found EBDC (ppm)		Average	*Corrected for Check	Avg. % Recovery	Corrected for Avg. % Recovery
Rep I Check	<0.1	<0.1	<0.1			
Rep II Check	0.1	<0.1	0.1			
Rep III Check	0.1	0.1	0.1			
Rep I 1x	0.3	0.3	0.3	0.3	81.3	0.4
Rep II 1x	0.3	0.3	0.3	0.2		0.2
Rep III 1x	0.3	0.3	0.3	0.2		0.2
Rep I 4x	0.4	0.4	0.4	0.4	81.3	0.5
Rep II 4x	0.6	0.6	0.6	0.5		0.6
Rep III 4x	0.4	0.4	0.4	0.3		0.4

Recovery Studies EBDC (ppm)

	Amount Added	Amount Found	*Corrected for Check	% Recovery	Avg. % Recovery
Rep I Check	1.2	1.0	1.0	83	
Rep II Check	1.2	1.0	0.9	75	
Rep III Check	1.2	1.0	0.9	83	
Rep I Check	3.0	3.0	3.0	100	
Rep II Check	3.0	3.2	3.1	79	
Rep III Check	3.0	2.1	2.0	67	81.3

*Corrected by subtraction of amount found in check sample.

Table II(A)-1-C. Residues of EBDC (from M-22) in
West Coast Canned Whole Tomatoes

Sample	Amount Found EBDC (ppm)		Average	*Corrected for Check	Avg. % Recovery	Corrected for Avg. % Recovery
Rep I Check	<0.1	<0.1	<0.1			
Rep II Check	0.1	0.1	0.1			
Rep III Check	<0.1	<0.1	<0.1			
Rep I 1x	0.2	0.3	0.3	0.3	67.5	0.4
Rep II 1x	0.1	0.2	0.2	0.1		0.1
Rep III 1x	0.2	0.2	0.2	0.2		0.3
Rep I 4x	0.3	0.4	0.4	0.4	67.5	0.6
Rep II 4x	0.2	0.2	0.2	0.1		0.1
Rep III 4x	0.3	0.3	0.3	0.3		0.4

Recovery Studies EBDC (ppm)

	Amount Added	Amount Found	*Corrected for Check	% Recovery	Avg. % Recovery
Rep I Check	1.2	0.9	0.9	75	
Rep II Check	1.2	0.9	0.8	67	
Rep III Check	1.2	0.9	0.9	75	
Rep I Check	3.0	2.0	2.0	67	
Rep II Check	3.0	2.0	1.9	63	
Rep III Check	3.0	2.0	2.0	67	67.5

*Corrected by subtraction of amount found in check sample.

Table IIA-1-D. Residues of EBDC (from M-22) in
West Coast Canned Whole Tomatoes Simmered for 5 Minutes

Sample	Amount Found EBDC (ppm)		Average	*Corrected for Check	Avg. % Recovery	Corrected for Avg. % Recovery
Rep I Check						
Rep II Check	<0.1	<0.1	<0.1			
Rep III Check	<0.1	<0.1	<0.1			
Rep I 1x	0.1	0.2	0.2	0.2	0.2	0.2
Rep II 1x	0.2	0.2	0.2	0.2	0.2	0.2
Rep III 1x	0.1	0.1	0.1	0.1	0.1	0.1
Rep I 4x	0.4	0.4	0.4	0.4	0.4	0.4
Rep II 4x	0.3	0.3	0.3	0.3	0.3	0.3
Rep III 4x	0.3	0.3	0.3	0.3	0.3	0.3

Recovery Studies EBDC (ppm)

	Amount Added	Amount Found	*Corrected for Check	% Recovery	Avg. % Recovery
Rep I Check					
Rep II Check	1.2 EBDC**	1.3	1.3	108	91
Rep III Check	1.2 EBDC**	1.3	1.3	108	
Rep I Check					
Rep II Check	3.1 EBDC**	2.6	2.6	84	84
Rep III Check	3.1 EBDC**	2.6	2.6	84	

*Corrected by subtraction of amount found in check sample.

Table IIA-1-E. Residues of EBDC (from M-22) in
West Coast Canned Whole Tomatoes Simmered for 60 Minutes

Sample	Amount Found EBDC (ppm)		Average	*Corrected for Check	Corrected for % Recovery
Rep I Check	<0.1	<0.1	<0.1		
Rep II Check	<0.1	<0.1	<0.1		
Rep III Check	<0.1	<0.1	<0.1		
Rep I 1x	0.1	0.1	0.1	0.1	0.1
Rep II 1x	0.2	0.2	0.2	0.2	0.2
Rep III 1x	0.1	0.1	0.1	0.1	0.1
Rep I 4x	0.2	0.2	0.2	0.2	0.2
Rep II 4x	0.2	0.3	0.3	0.3	0.4
Rep III 4x	0.3	0.3	0.3	0.3	0.4

Recovery Studies EBDC (ppm)

	Amount Added	Amount Found	*Corrected for Check	% Recovery	Avg. % Recovery
Rep I Check	1.2 EBDC**	1.1	1.1	92	83.9
Rep II Check	0.6 EBDC***	0.6	0.6	100	
Rep III Check	0.6 EBDC***	0.4	0.4	67	
Rep I Check	3.1 EBDC**	2.9	2.9	94	88
Rep II Check	1.6 EBDC***	1.4	1.4	88	
Rep III Check	1.6 EBDC***	0.9	0.9	56	

*Corrected by subtraction of amount found in check sample.

**Amount added has been corrected for % activity of 60.

***Amount added has been corrected for % activity of 30. EBDC in fortified starch had suddenly degraded.

Table IIA-1-F. Residues of EBDC (from M-22) in
West Coast Tomato Juice

Sample	Amount Found EBDC (ppm)		Avg.	*Corrected for Check	Avg. % Recovery	Corrected for Avg. % Recovery
Rep I Check	<0.1	<0.1	<0.1			
Rep II Check	<0.1	<0.1	<0.1			
Rep III Check	<0.1	<0.1	<0.1			
Rep I 1x	0.3	0.2	0.3	0.3	67	0.4
Rep II 1x	0.2	0.3	0.3	0.3		0.4
Rep III 1x	0.2	0.2	0.2	0.2		0.3
Rep I 4x	0.6	0.6	0.6	0.6	67	0.9
Rep II 4x	0.4	0.4	0.4	0.4		0.6
Rep III 4x	0.7	0.7	0.7	0.7		1.0

Recovery Studies EBDC (ppm)

	Amount Added	Amount Found	*Corrected for Check	% Recovery	Avg. % Recovery
Rep I Check	1.2	0.9	0.9	75	
Rep II Check	1.2	0.8	0.8	67	
Rep III Check	1.2	0.9	0.9	75	
Rep I Check	3.0	1.8	1.8	60	
Rep II Check	3.0	2.0	2.0	67	
Rep III Check	3.0	2.0	2.0	67	67

*Corrected by subtraction of amount found in check sample.

Table IIA-1-G. Residues of EBDC (from M-22) in
West Coast Tomato Sauce

Sample	Amount Found EBDC (ppm)		Average	*Corrected for Check	Corrected for % Recovery
Rep I Check	<0.1				
Rep II Check	<0.1				
Rep III Check	<0.1				
Rep I 1x	0.8	0.8	0.8	0.8	0.7
Rep II 1x	0.9	0.9	0.9	0.9	0.8
Rep III 1x	1.0	1.0	1.0	1.0	0.9
Rep I 4x	2.3	2.3	2.3	2.3	2.0
Rep II 4x	2.6	2.6	2.6	2.6	2.5
Rep III 4x	2.7	2.0	2.9	2.9	2.5

Recovery Studies EBDC (ppm)

	Amount Added	Amount Found	*Corrected for Check	% Recovery	Avg. % Recovery
Rep I Check	1.2 EBDC**	1.0	1.0	100	114
Rep II Check	1.2 EBDC**	1.4	1.4	117	
Rep III Check	1.2 EBDC**	0.9	0.9	75	

*Corrected by subtraction of amount found in check sample.

**Amount added has been corrected for % activity of 60.

Table IIA-1-H. Residues of EBDC (from M-22) in
West Coast Tomato Paste

<u>Sample</u>	<u>Amount Found EBDC (ppm)</u>		<u>Average</u>	<u>*Corrected for Check</u>	<u>Corrected for % Recovery</u>
Rep I Check	0.1				
Rep II Check	<0.1				
Rep III Check	0.1				
Rep I 1x	1.7	1.7	1.7	1.6	2.1
Rep II 1x	1.7	1.7	1.7	1.7	2.2
Rep III 1x	1.9	1.9	1.9	1.8	2.3
Rep I 4x	4.1	3.0	3.6	3.6	4.6
Rep II 4x	2.7	2.7	2.7	2.7	3.6
Rep III 4x	2.6	2.6	2.6	2.6	3.2

Recovery Studies EBDC (ppm)

	<u>Amount Added</u>	<u>Amount Found</u>	<u>*Corrected for Check</u>	<u>% Recovery</u>	<u>Avg. % Recovery</u>
Rep I Check	1.2 EBDC**	1.0	0.0	75	78
Rep II Check	1.2 EBDC**	1.0	1.0	83	
Rep III Check	1.2 EBDC**	1.0	0.9	75	

*Corrected by subtraction of amount found in check sample.

**Amount added has been corrected for % activity of 80.

Table IIA-2-A. Residues of ETU (from M-22) in
West Coast Unwashed Tomatoes

Sample	Amount Found EBOC (ppm)		Average	*Corrected for Check	Corrected for % Recovery
Rep I Check	0.01	0.01	0.01		
Rep II Check	0.02	0.01	0.02		
Rep III Check	0.01	0.01	0.01		
Rep I 1x	0.02	<0.01	0.01	<0.01	<0.01
Rep II 1x	0.01	0.02	0.02	<0.01	<0.01
Rep III 1x	0.03	0.01	0.02	0.01	0.01
Rep I 4x	<0.01	0.02	0.01	<0.01	<0.01
Rep II 4x	0.03	0.04	0.04	0.02	0.03
Rep III 4x	0.04	0.03	0.04	0.03	0.04

Recovery Studies ETU (ppm)

	Amount Added	Amount Found	*Corrected for Check	% Recovery	Avg. % Recovery
Rep I Check	0.05 ETU	0.04	0.03	60	67
Rep II Check	0.05 ETU	0.06	0.04	80	
Rep III Check	0.05 ETU	0.04	0.03	60	

*Corrected by subtraction of amount found in check sample.

Table IIA-2-B. Residues of ETU (from M-22) in
West Coast Washed Tomatoes

Sample	Amount Found SBOC (ppm)		Average	*Corrected for Check	Corrected for % Recovery
Rep I Check	0.01				
Rep II Check	0.01	0.03	0.02		
Rep III Check	0.02	0.01	0.02		
Rep I 1x	0.01	0.01	0.01	<0.01	<0.01
Rep II 1x	0.01	0.01	0.01	<0.01	<0.01
Rep III 1x	0.01	0.01	0.01	<0.01	<0.01
Rep I 4x	0.07	0.10	0.08	0.07	0.12
Rep II 4x	0.07	0.16	0.11	0.09	0.14
Rep III 4x	0.03	0.02	0.03	0.01	0.01

Recovery Studies ETU (ppm)

	Amount Added	Amount Found	*Corrected for Check	% Recovery	Avg. % Recovery
Rep I Check	0.05 ETU	0.06	0.05	100	67
Rep II Check	0.05 ETU	0.04	0.02	40	
Rep III Check	0.05 ETU	0.04	0.02	40	

*Corrected by subtraction of amount found in check sample.

Table IIA-2-C. Residues of ETU (from M-22) in
West Coast Canned Whole Tomatoes

<u>Sample</u>	<u>Amount Found EBOC (ppm)</u>		<u>Average</u>	<u>*Corrected for Check</u>	<u>Corrected for % Recovery</u>
Rep I Check	0.02	0.01	0.02		
Rep II Check	<0.01	0.01	0.01		
Rep III Check	0.01	0.02	0.01		
Rep I 1x	0.02	0.03	0.03	0.01	0.01
Rep II 1x	0.02	0.05	0.04	0.03	0.04
Rep III 1x	0.01	0.01	0.01	<0.01	<0.01
Rep I 4x	0.07	0.08	0.08	0.06	0.09
Rep II 4x	0.04	0.04	0.04	0.03	0.04
Rep III 4x	0.07	0.07	0.07	0.06	0.09

Recovery Studies ETU (ppm)

	<u>Amount Added</u>	<u>Amount Found</u>	<u>*Corrected for Check</u>	<u>% Recovery</u>	<u>Avg. % Recovery</u>
Rep I Check	0.01 ETU	0.04	0.02	40	67
Rep II Check	0.05 ETU	0.06	0.05	100	
Rep III Check	0.05 ETU	0.04	0.03	60	

*Corrected by subtraction of amount found in check sample.

Table IIA-2-D. Residues of ETU (from M-22) in
West Coast Whole Tomatoes Simmered for 5 Minutes

Sample	Amount Found EBDC (ppm)		Average	*Corrected for Check	Corrected for % Recovery
Rep I Check	<0.01		<0.01		
Rep II Check	<0.01	<0.01	<0.01		
Rep III Check	<0.01	<0.01	<0.01		
Rep I 1x	0.04	0.03	0.04	0.04	0.06
Rep II 1x	0.03	0.12	0.08	0.08	0.10
Rep III 1x	0.03	0.03	0.03	0.03	0.04
Rep I 4x	0.06	0.06	0.06	0.06	0.08
Rep II 4x	0.03	0.03	0.03	0.03	0.04
Rep III 4x	0.04	0.04	0.04	0.04	0.06

Recovery Studies ETU (ppm)

	Amount Added	Amount Found	*Corrected for Check	% Recovery	Avg. % Recovery
Rep I Check	0.05 ETU	0.03	0.03	60	80
Rep II Check	0.05 ETU	0.05	0.05	100	
Rep III Check	0.05 ETU	0.02	0.02	40	

*Corrected by subtraction of amount found in check sample.

Table IIA-2-E. Residues of ETU (from M-22) in
West Coast Canned Whole Tomatoes Simmered for 60 minutes

Sample	Amount Found EBOC (ppm)		Average	*Corrected for Check	Corrected for % Recovery
Rep I Check	<0.01	<0.01	<0.01		
Rep II Check	<0.01		<0.01		
Rep III Check	<0.01		<0.01		
Rep I 1x	0.06		0.06	0.06	0.12
Rep II 1x	0.04	0.03	0.04	0.04	0.08
Rep III 1x	0.01	0.01	0.01	0.01	0.02
Rep I 4x	0.06	0.06	0.06	0.06	0.12
Rep II 4x	0.07	0.05	0.06	0.06	0.12
Rep III 4x	0.04	0.06	0.05	0.05	0.10

Recovery Studies ETU (ppm)

	Amount Added	Amount* Found	*Corrected for Check	% Recovery	Avg. % Recovery
Rep I Check	0.05 ETU	0.03	0.03	60	60
Rep II Check	0.05 ETU	0.02	0.02	40	
Rep III Check	0.05 ETU	0.02	0.02	40	

*Corrected by subtraction of amount found in check sample.

Table IIA-2-F. Residues of ETU (from M-22) in
West Coast Tomato Juice

Sample	Amount Found EBOC (ppm)		Average	*Corrected for Check	Corrected for % Recovery
Rep I Check	0.01	0.02	0.01		
Rep II Check	0.02	0.02	0.02		
Rep III Check	0.02	0.03	0.03		
Rep I 1x	0.05	0.05	0.05	0.04	0.07
Rep II 1x	0.04	0.05	0.05	0.03	0.05
Rep III 1x	0.03	0.03	0.03	<0.01	<0.01
Rep I 4x	0.05	0.04	0.05	0.04	0.07
Rep II 4x	0.11	0.12	0.12	0.10	0.17
Rep III 4x	0.11	0.11	0.11	0.08	0.13

Recovery Studies ETU (ppm)

	Amount Added	Amount Found	*Corrected for Check	% Recovery	Avg. % Recovery
Rep I Check	0.05 ETU	0.05	0.04	80	60
Rep II Check	0.05 ETU	0.05	0.03	60	
Rep III Check	0.05 ETU	0.05	0.02	40	

*Corrected by subtraction of amount found in check sample.

Table IIA-2-0. Residues of ETU (from H-22) in
West Coast Tomato Sauce

Sample	Amount Found EBOC (ppm)		Average	*Corrected for Check	Corrected for % Recovery
Rep I Check	0.09		0.09		
Rep II Check	0.04		0.04		
Rep III Check	0.04		0.04		
Rep I 1x	0.23	0.26	0.24	0.16	0.20
Rep II 1x	0.01	0.08	0.06	0.01	0.01
Rep III 1x	0.06	0.12	0.09	0.06	0.12
Rep I 4x	0.26	0.29	0.27	0.18	0.25
Rep II 4x	0.12	0.21	0.17	0.13	0.18
Rep III 4x	0.19	0.20	0.20	0.16	0.22

Recovery Studies ETU (ppm)

	Amount Added	Amount Found	*Corrected for Check	% Recovery	Avg. % Recovery
Rep I Check	0.06 ETU	0.16	0.06	120	73.3
Rep II Check	0.05 ETU	0.06	0.02	40	
Rep III Check	0.06 ETU	0.07	0.03	60	

*Corrected by subtraction of amount found in check sample.

Table IIA-2-H. Residues of ETU (from M-22) in
West Coast Tomato Paste

Sample	Amount Found EBOC (ppm)		Average	*Corrected for Check	Corrected for % Recovery
Rep I Check	0.03	0.03	0.03		
Rep II Check	0.03		0.03		
Rep III Check	0.06		0.06		
Rep I 1x	0.38	0.35	0.37	0.34	0.72
Rep II 1x	0.15	0.24	0.20	0.17	0.36
Rep III 1x	0.06	0.06	0.06	0.01	0.02
Rep I 4x	0.16	0.40	0.28	0.25	0.53
Rep II 4x	0.26	0.07	0.18	0.15	0.32
Rep III 4x	0.26	0.34	0.30	0.25	0.53

Recovery Studies ETU (ppm)

	Amount Added	Amount Found	*Corrected for Check	% Recovery	Avg. % Recovery
Rep I Check	0.06 ETU	0.06	0.03	60	47
Rep II Check	0.06 ETU	0.06	0.02	40	
Rep III Check	0.06 ETU	0.07	0.02	40	

*Corrected by subtraction of amount found in check sample.

Table IIB-1-A. Residues of EBDC (from M-22) in
West Coast Unwashed Spinach

Sample	Amount Found EBDC (ppm)		Average	*Corrected for Check	Corrected for % Recovery
Rep I Check	0.5	0.5	0.5		
Rep II Check	0.2	0.2	0.2		
Rep III Check	<0.1	<0.1	<0.1		
Rep I 1x	58.3	59.6	59.0	58.5	78.0
Rep II 1x	70.1	71.7	70.9	70.7	91.8
Rep III 1x	67.8	70.6	69.2	69.2	89.9
Rep I 4x	119.3	123.4	121.4	120.9	157.0
Rep II 4x	155.6	158.9	157.3	157.1	204.0
Rep III 4x	151.1	154.4	152.8	152.8	199.4

Recovery Studies ETU (ppm)

	Amount Added	Amount Found	*Corrected for Check	% Recovery	Avg. % Recovery
Rep I Check	1.5 EBDC**	1.6	1.1	73	77
Rep II Check	1.2 EBDC**	1.0	0.8	67	
Rep III Check	1.2 EBDC**	1.2	1.2	100	
Rep I Check	7.5 EBDC**	6.3	5.8	77	77
Rep II Check	3.1 EBDC**	2.2	2.0	65	
Rep III Check	3.1 EBDC**	2.7	2.7	77	

*Corrected by subtraction of amount found in check sample.

**Amount added has been corrected for % activity of 60.

Table 11B-1-B: Residues of EBDC (from M-22) in
West Coast Washed Spinach

Sample	Amount Found EBDC (ppm)		Average	*Corrected for Check	Corrected for % Recovery
Rep I Check	0.2	0.2			
Rep II Check	<0.1	<0.1			
Rep III Check	<0.1	<0.1			
Rep I 1x	26.2	26.9	26.6	26.4	36.2
Rep II 1x	25.4	25.6	25.5	25.5	34.9
Rep III 1x	26.6	26.6	26.1	26.1	34.4
Rep I 4x	104.1	106.2	105.2	105.0	143.6
Rep II 4x	157.2	164.4	155.8	155.8	213.4
Rep III 4x	155.6	165.4	160.6	160.6	220.0

Recovery Studies EBDC (ppm)

	Amount Added	Amount Found	*Corrected for Check	% Recovery	Avg. % Recovery
Rep I Check	1.5 EBDC**	1.3	1.1	73	73
Rep II Check	1.2 EBDC**	1.0	1.0	83	
Rep III Check	1.2 EBDC**	1.0	1.0	83	
Rep I Check	7.5 EBDC**	6.3	6.1	81	87
Rep II Check	3.1 EBDC**	2.2	2.2	71	
Rep III Check	3.1 EBDC**	2.7	2.7	87	

*Corrected by subtraction of amount found in check sample.

**Amount added has been corrected for % activity of 60.

Table 11B-1-C. Residues of EBDC (from M-22) in
West Coast Frozen Blanched Spinach

Sample	Amount Found EBDC (ppm)		Average	*Corrected for Check	Corrected for % Recovery
Rep I Check	<0.1	<0.1	<0.1		
Rep II Check	<0.1	<0.1	<0.1		
Rep III Check	<0.1	<0.1	<0.1		
Rep I 1x	0.3	0.3	0.3	0.3	0.4
Rep II 1x	0.2	0.2	0.2	0.2	0.3
Rep III 1x	0.4	0.4	0.4	0.4	0.6
Rep I 4x	0.6	0.6	0.6	0.6	0.9
Rep II 4x	0.5	0.5	0.5	0.5	0.7
Rep III 4x	0.8	0.8	0.8	0.8	1.1

Recovery Studies EBDC (ppm)

	Amount Added	Amount Found	*Corrected for Check	% Recovery	Avg. % Recovery
Rep I Check	1.2 EBDC**	0.9	0.9	75	70
Rep II Check	1.2 EBDC**	0.9	0.9	75	
Rep III Check	1.2 EBDC**	0.9	0.9	75	
Rep I Check	3.1 EBDC**	2.1	2.1	68	68
Rep II Check	3.1 EBDC**	2.1	2.1	68	
Rep III Check	3.1 EBDC**	2.1	2.1	68	

*Corrected by subtraction of amount found in check sample.

**Amount added has been corrected for % activity of 60.

Table IIB-1-D. Residues of EBDC (from M-22) in
West Coast Frozen Blanched Spinach Simmered for 5 Minutes

Sample	Amount Found EBDC (ppm)		Average	*Corrected for Check	Corrected for % Recovery
Rep I Check	<0.1	<0.1	<0.1		
Rep II Check	<0.1	<0.1	<0.1		
Rep III Check	<0.1	<0.1	<0.1		
Rep I 1x	0.1	0.1	0.1	0.1	0.1
Rep II 1x	0.2	0.2	0.2	0.2	0.3
Rep III 1x	0.1	0.1	0.1	0.1	0.1
Rep I 4x	0.3	0.3	0.3	0.3	0.4
Rep II 4x	0.3	0.3	0.3	0.3	0.4
Rep III 4x	0.3	0.3	0.3	0.3	0.4

Recovery Studies EBDC (ppm)

	Amount Added	Amount Found	*Corrected for Check	% Recovery	Avg. % Recovery
Rep I Check	1.2 EBDC**	0.9	0.9	75	75
Rep II Check	1.2 EBDC**	0.9	0.9	75	
Rep III Check	1.2 EBDC**	0.9	0.9	75	

*Corrected by subtraction of amount found in check sample.

**Amount added has been corrected for % activity of 60.

Table IIb-1-E. Residues of EBDC (from M-12) in
Drained Water from West Coast Frozen Blanched Spinach
Simmered for 5 Minutes

Sample	Amount Found EBDC (ppm)		Average	*Corrected for Check	Corrected for % Recovery
Rep I Check	<0.1	<0.1			
Rep II Check	<0.1	<0.1			
Rep III Check	<0.1	<0.1			
Rep I 1x	<0.1	<0.1	<0.1	<0.1	<0.1
Rep II 1x	<0.1	<0.1	<0.1	<0.1	<0.1
Rep III 1x	<0.1	<0.1	<0.1	<0.1	<0.1
Rep I 4x	<0.1	<0.1	<0.1	<0.1	<0.1
Rep II 4x	<0.1	<0.1	<0.1	<0.1	<0.1
Rep III 4x	<0.1	<0.1	<0.1	<0.1	<0.1

Recovery Studies EBDC (ppm)

	Amount Added	Amount Found	*Corrected for Check	% Recovery	Avg. % Recovery
Rep I Check	1.2 EBDC**	0.9	0.9	75	75
Rep II Check	1.2 EBDC**	0.9	0.9	75	
Rep III Check	1.2 EBDC**	0.9	0.9	75	

*Corrected by subtraction of amount found in check sample.

**Amount added has been corrected for % activity of 60.

Table IIB-1-F. Residues of EBDC (from H-22) from
West Coast Canned Spinach

Sample	Amount Found EBDC (ppm)		Average	*Corrected for Check	Corrected for % Recovery
Rep I Check	≤0.1	≤0.1	≤0.1		
Rep II Check	≤0.1	≤0.1	≤0.1		
Rep III Check	≤0.1	≤0.1	≤0.1		
Rep I 1x	≤0.1	≤0.1	≤0.1	≤0.1	≤0.1
Rep II 1x	≤0.1	≤0.1	≤0.1	≤0.1	≤0.1
Rep III 1x	≤0.1	≤0.1	≤0.1	≤0.1	≤0.1
Rep I 4x	≤0.1	≤0.1	≤0.1	≤0.1	≤0.1
Rep II 4x	≤0.1	≤0.1	≤0.1	≤0.1	≤0.1
Rep III 4x	≤0.1	≤0.1	≤0.1	≤0.1	≤0.1

Recovery Studies EBDC (ppm)

	Amount Added	Amount Found	*Corrected for Check	% Recovery	Avg. % Recovery
Rep I Check	1.2 EBDC**	0.9	0.9		67
Rep II Check	1.2 EBDC**	0.9	0.9		
Rep III Check	1.2 EBDC**	0.9	0.9		
Rep I Check	3.1 EBDC**	1.8	1.8		
Rep II Check	3.1 EBDC**	2.1	2.1		
Rep III Check	3.1 EBDC**	2.0	2.0		

*Corrected by subtraction of amount found in check sample.

**Amount added has been corrected for % activity of 60.

Table 11B-1-G. Residues of EBDC (from M-22) in Canned
West Coast Spinach Cooked for 5 Minutes

Sample	Amount Found EBDC (ppm)		Average	*Corrected for Check	Corrected for % Recovery
Rep I Check	0.1	0.1	0.1		
Rep II Check	0.1	0.1	0.1		
Rep III Check	<0.1	<0.1	<0.1		
Rep I 1x	<0.1	<0.1	<0.1	<0.1	<0.1
Rep II 1x	<0.1	<0.1	<0.1	<0.1	<0.1
Rep III 1x	<0.1	<0.1	<0.1	<0.1	<0.1
Rep I 4x	0.1	0.1	<0.1	<0.1	<0.1
Rep II 4x	0.1	0.1	<0.1	<0.1	<0.1
Rep III 4x	<0.1	<0.1	<0.1	<0.1	<0.1

Recovery Studies EBDC (ppm)

	Amount Added	Amount Found	*Corrected for Check	% Recovery	Avg. % Recovery
Rep I Check	1.2 EBDC**	0.9	0.6	67	69
Rep II Check	1.2 EBDC**	0.9	0.6	67	
Rep III Check	1.2 EBDC**	0.9	0.9	75	

*Corrected by subtraction of amount found in check sample.

**Amount added has been corrected for % activity of 60.

Table IIB-1-H. Residues of EBDC (from M-22) in
Drained Water from West Coast Canned Spinach
Cooked for 5 Minutes

Sample	Amount Found EBDC (ppm)		Average	*Corrected for Check	Corrected for % Recovery
Rep I Check	<0.1	<0.1	<0.1		
Rep II Check	<0.1	<0.1	<0.1		
Rep III Check	<0.1	<0.1	<0.1		
Rep I 1x	<0.1	<0.1	<0.1		
Rep II 1x	0.7	0.7	0.7		<0.1 ¹
Rep III 1x	<0.1	<0.1	<0.1		0.9 ¹
					<0.1
Rep I 4x	<0.1	<0.1	<0.1		
Rep II 4x	<0.1	<0.1	<0.1		<0.1
Rep III 4x	<0.1	<0.1	<0.1		<0.1
					<0.1

Recovery Studies EBDC (ppm)

	Amount Added	Amount Found	*Corrected for Check	% Recovery	Avg. % Recovery
Rep I Check	1.2 EBDC**	0.9	0.9	75	75
Rep II Check	1.2 EBDC*	0.9	0.9	75	
Rep III Check	1.2 EBDC**	0.9	0.9	75	

*Corrected by subtraction of amount found in check sample.

**Amount added has been corrected for % activity of 60.

¹Value excluded from summary table.

Table IIB-2-A. Residues of ETU (from M-22) in
West Coast Unwashed Spinach

Sample	Amount Found EBOC (ppm)		Average	*Corrected for Check	Corrected for % Recovery
Rep I Check	0.02	<0.01	0.01		
Rep II Check	<0.01	<0.01	<0.01		
Rep III Check	<0.01	<0.01	<0.01		
Rep I 1x	0.13	0.08	0.21	0.20	0.38
Rep II 1x	0.03	0.04	0.04	0.04	0.08
Rep III 1x	0.07	0.18	0.13	0.13	0.25
Rep I 4x	0.21	0.18	0.20	0.19	0.36
Rep II 4x	0.69	0.52	0.61	0.61	1.18
Rep III 4x	0.18	0.53	0.36	0.36	0.68

Recovery Studies ETU (ppm)

	Amount Added	Amount Found	*Corrected for Check	% Recovery	Avg. % Recovery
Rep I Check	0.05 ETU	0.04			
Rep II Check	0.05 ETU	0.03	0.03	60	63
Rep III Check	0.05 ETU	0.02	0.03	60	
			0.02	40	

*Corrected by subtraction of amount found in check sample.

Table IIB-2-B. Residues of ETU (From H-22) in
West Coast Washed Spinach

Sample	Amount Found EDDC (ppm)		Average	*Corrected for Check	Corrected for % Recovery
Rep I Check	<0.01	<0.01	<0.01		
Rep II Check	0.01	<0.01	<0.01		
Rep III Check	<0.01	<0.01	<0.01		
Rep I 1x	0.03	0.03	0.03	0.03	0.06
Rep II 1x	0.04	0.04	0.04	0.04	0.08
Rep III 1x	0.03	<0.01	0.02	0.02	0.04
Rep I 4x	0.01	0.01	0.01	0.01	0.02
Rep II 4x	0.44	0.67	0.57	0.57	1.08
Rep III 4x	0.08	0.06	0.07	0.07	0.13

Recovery Studies ETU (ppm)

	Amount Added	Amount Found	*Corrected for Check	% Recovery	Avg. % Recovery
Rep I Check	0.05 ETU	0.03	0.03	60	53
Rep II Check	0.05 ETU	0.02	0.02	40	
Rep III Check	0.05 ETU	0.03	0.03	60	

*Corrected by subtraction of amount found in check sample.

**Amount added has been corrected for % activity of e_0 .

Table 11B-2-C. Residues of ETU (from M-22) in
West Coast Frozen Blanched Spinach

Sample	Amount Found EBOC (ppm)		Average	*Corrected for Check	Corrected for % Recovery
Rep I Check	0.01	0.01	0.01		
Rep II Check	0.01	0.01	0.01		
Rep III Check	<0.01	<0.01	<0.01		
Rep I 1x	0.37	0.34	0.36	0.36	0.88
Rep II 1x	0.46	0.55	0.51	0.50	1.26
Rep III 1x	0.34	0.09	0.22	0.22	0.56
Rep I 4x	0.65	0.17	0.41	0.40	1.00
Rep II 4x	0.36	1.42	0.89	0.88	2.20
Rep III 4x	0.90	1.33	1.12	1.12	2.80

Recovery Studies ETU (ppm)

	Amount Added	Amount Found	*Corrected for Check	% Recovery	Avg. % Recovery
Rep I Check	0.05 ETU	0.03	0.02	40	40
Rep II Check	0.05 ETU	0.03	0.02	40	
Rep III Check	0.05 ETU	0.02	0.02	40	

*Corrected by subtraction of amount found in check sample.

Table 11B-2-D. Residues of ETU (from M-22) in
West Coast Frozen Blanched Spinach
Simmered for 6 Minutes

Sample	Amount Found EDC (ppm)		Average	*Corrected for Check	Corrected for % Recovery
Rep I Check	0.01				
Rep II Check	<0.01				
Rep III Check	<0.01		<0.01		
Rep I 1x	0.66	0.25	0.46	0.44	1.10
Rep II 1x	0.62	0.34	<0.40	0.47	1.18
Rep III 1x	<0.01	<0.01	<0.01	0.01	0.01
Rep I 4x	0.71	0.70	0.71	0.70	1.75
Rep II 4x	0.68	0.62	0.66	0.66	1.63
Rep III 4x	9.30	0.48	0.39	0.38	0.95

Recovery Studies ETU (ppm)

	Amount Added	Amount Found	*Corrected for Check	% Recovery	Avg. % Recovery
Rep I Check	0.05 ETU	0.03	0.02	40	40
Rep II Check	0.05 ETU	0.02	0.02	40	
Rep III Check	0.05 ETU	0.02	0.02	40	

*Corrected by subtraction of amount found in check sample.

**Amount added has been corrected for % activity of 60.

Table IIB-2-E. Residues of ETU (from M-22) in
Drained Water from West Coast Frozen Blanched Spinach
Simmered for 5 Minutes

Sample	Amount Found EBDC (ppm)		Average	*Corrected for Check	Corrected for % Recovery
Rep I Check	<0.01				
Rep II Check	0.01				
Rep III Check	<0.01				
Rep I 1x	0.22	0.40	0.31	0.31	0.52
Rep II 1x	0.30	0.32	0.31	0.31	0.52
Rep III 1x	0.59	0.51	0.55	0.55	0.92
Rep I 4x	0.55	0.55	0.55	0.55	0.92
Rep II 4x	0.34	0.59	0.47	0.47	0.78
Rep III 4x	0.01	0.02	0.02	0.02	0.03

Recovery Studies ETU (ppm)

	Amount Added	Amount Found	*Corrected for Check	% Recovery	Avg. % Recovery
Rep I Check	0.05 ETU	0.03	0.03	60	60
Rep II Check	0.05 ETU	0.03	0.03	60	
Rep III Check	0.05 ETU	0.03	0.03	60	

*Corrected by subtraction of amount found in check sample.

Table 11B-2-F. Residues of ETU (from M-22) in
West Coast Canned Spinach

Sample	Amount Found EBDC (ppm)		Average	*Corrected for Check	Corrected for % Recovery
Rep I Check	0.01	0.01	0.01		
Rep II Check	0.01	0.01	0.01		
Rep III Check	0.01	0.01	0.01		
Rep I 1x	0.67	0.66	0.67	0.66	1.65
Rep II 1x	0.78	0.78	0.78	0.77	1.93
Rep III 1x	0.78	0.73	0.76	0.76	1.00
Rep I 4x	1.29	0.61	0.95	0.94	2.38
Rep II 4x	1.31	1.64	1.43	1.42	3.55
Rep III 4x	0.83	0.47	0.65	0.64	1.60

Recovery Studies ETU (ppm)

	Amount Added	Amount Found	*Corrected for Check	% Recovery	Avg. % Recovery
Rep I Check	0.06 ETU	0.03	0.02	40	40
Rep II Check	0.06 ETU	0.03	0.02	40	
Rep III Check	0.06 ETU	0.03	0.02	40	

*Corrected by subtraction of amount found in check sample.

Table 11B-2-G. Residues of ETU (from M-22) in
West Coast Canned Spinach Cooked for 5 Minutes

Sample	Amount Found	EBDC (ppm)	Average	*Corrected for Check	Corrected for % Recovery
Rep I Check		0.01			
Rep II Check		0.01			
Rep III Check		0.01			
Rep I 1x	0.33	0.36	0.36	0.34	0.85
Rep II 1x	1.33	0.70	1.02	1.01	2.53
Rep III 1x	1.16	1.23	1.20	1.19	2.98
Rep I 4x	1.13	1.08	1.11	1.10	2.75
Rep II 4x	1.26	1.39	1.33	1.32	3.30
Rep III 4x	0.31	0.29	0.30	0.29	0.73

Recovery Studies ETU (ppm)

	Amount Added	Amount Found	*Corrected for Check	% Recovery	Avg. % Recovery
Rep I Check	.05 ETU	0.03	0.02	40	40
Rep II Check	0.05 ETU	0.03	0.02	40	
Rep III Check	0.05 ETU	0.03	0.02	40	

*Corrected by subtraction of amount found in check sample.

Table IIB-2-H. Residues of ETU (from M-22) in
Drained Water From West Coast Canned Spinach

<u>Sample</u>	<u>Amount Found EBDC (ppm)</u>	<u>Average</u>	<u>*Corrected for Check</u>	<u>Corrected for % Recovery</u>
Rep I check	<0.01			
Rep II check	<0.01			
Rep III check	<0.01			
Rep I 1x	0.59 0.14	0.37	0.37	6.70
Rep II 1x	1.11 1.09	1.10	1.10	2.08
Rep III 1x	0.12 0.19	0.16	0.16	0.30
Rep I 4x	0.25 0.23	0.24	0.24	0.45
Rep II 4x	0.28 0.26	0.27	0.27	0.51
Rep III 4x	0.54 0.27	0.41	0.41	0.77

Recovery Studies ETU (ppm)

	<u>Amount Added</u>	<u>Amount Found</u>	<u>*Corrected for Check</u>	<u>% Recovery</u>	<u>Average % Recovery</u>
Rep I check	0.05 ETU	.03	0.03	60	53
Rep II check	0.05 ETU	0.02	0.02	40	
Rep III check	0.05 ETU	.03	0.03	60	

*Corrected by subtraction of amount found in check sample.

APPENDIX D

Tables III to VIII
Additional Summary and Individual-Data Tables

Table III. Effects of Washing and Canning on EBDC and ETU Residues
in Tomatoes, Carrots, and Spinach Treated with Dithane M-45^(a)

Commodity	Unwashed		Washed		% EBDC Removed	Canned		Conversion to ETU in Canning*
	EBDC	ETU	EBDC	ETU		EBDC	ETU	
East Coast Tomatoes								
Check	<0.1	<0.01	0.1	0.01	--	0.1	0.01	
1x	0.3	<0.01	0.2	<0.01	33	<0.1	0.03	40.8
4x	0.8	0.02	0.3	0.01	50	0.1	0.10	100.0
								Avg. 70.4
West Coast Tomatoes								
Check	<0.1	<0.01	<0.1	<0.01	--	0.1	0.01	
1x	2.1	0.01	0.6	0.01	76	0.5	0.11	30.9
4x	7.1	0.02	0.9	0.01	97	1.8	0.17	25.2
								Avg. 28.1
East Coast Carrots								
Check	<0.1	<0.01	<0.1	<0.01	--	<0.1	<0.01	
1x	0.6	<0.01	0.3	<0.01	80	<0.1	0.03	20.6
4x	0.8	0.01	0.7	<0.01	13	<0.1	0.05	14.7
								Avg. 17.7
West Coast Carrots								
Check	<0.1	<0.01	<0.1	<0.01	--	<0.1	<0.01	
1x	0.1	0.01	0.1	<0.01	none	0.1	<0.01	N.D.
4x	0.4	<0.01	0.4	<0.01	none	0.1	0.01	5.1
								Avg. 2.6
East Coast Spinach								
Check	0.2	<0.01	0.1	<0.01	--	0.1	0.01	
1x	2.5	<0.01	1.5	<0.01	40	<0.1	0.10	24.5
4x	6.6	0.01	2.7	0.01	59	<0.1	0.44	33.5
								Avg. 29.0
West Coast Spinach								
Check	0.3	0.01	0.2	<0.01	--	0.1	0.01	
1x	61.9	0.34	9.7	0.02	<	0.71		14.7
4x	219.3	1.41	58.7	0.08	73	0.1	2.66	9.0
								Avg. 11.9

*Calculations: EBDC wash (1x or 4x) = EBDC wash (check) x 0.486 = A
ETU canned (1x or 4x) = [ETU canned (check) + ETU wash
(1x or 4x)] = B

Conversion due to processing = B/A

Example:

East Coast tomatoes 1x

A = (0.2 - 0.1) 0.486 = 0.049

B = 0.03 = (0.01 + <0.01) = 0.02

B/A = $\frac{0.02}{0.049}$ = 0.408 x 100 = 40.8%

Table IV. Conversion of EBDC¹ (Added as Dithane M-45^(R))
to ETU on Control Samples of Tomatoes, Carrots,
and Spinach--at Various Processing Stages

Commodity (Location)	Process Stage	Replicate	ETU Found	ETU in check + 0.05 ETU	ETU from EBDC	% Conversion of EBDC to ETU
Tomatoes (East Coast)	Unwashed	I	0.19	0.07	0.12	20.1
		II	0.07	0.03	0.04	6.9
		III	0.04	0.03	0.01	1.7
	Washed	I	0.04		0.04	6.9
		II	0.11	0.04	0.07	11.1
		III	0.11	0.03	0.08	13.8
	Frozen	I	0.10	0.04	0.06	10.3
		II	0.08	0.01	0.07	12.1
		III	0.15	0.02	0.13	22.4
	Canned	I	0.17	0.05	0.12	20.1
		II	0.20	0.07	0.13	22.4
		III	0.32	0.05	0.27	46.6
	Avg. 16.3					
Tomatoes (West Coast)	Unwashed	I	0.17	0.07	0.14	24.1
		II	0.10	0.05	0.05	8.6
		III	0.11	0.03	0.06	10.3
	Washed	I	0.17	0.04	0.13	22.4
		II	0.09	0.01	0.08	13.8
		III	0.06	0.04	0.02	3.4
	Frozen	I	0.10	0.04	0.06	10.3
		II	0.28	0.05	0.23	40.0
		III	0.04	0.01	0.03	5.2
	Canned	I	0.08	0.03	0.05	8.6
		II	0.16	0.06	0.10	17.2
		III	0.18	0.02	0.16	27.6
	Avg. 16.0					

Table IV. (Continued)

Commodity (Location)	Process Stage	Replicate	ETU Found	ETU in check + 0.05 ETU	ETU from EMDC	% Conversion of EMDC to ETU
Carrots (East Coast)	Unwashed	I	0.12	0.05	0.07	12.1
		II	0.13	0.04	0.09	16.6
		III	0.11	0.03	0.08	13.8
	Washed	I	0.13	0.04	0.09	16.6
		II	0.14	0.03	0.11	19.0
		III	0.14	0.04	0.10	17.2
	Diced	I	0.10	0.03	0.07	12.1
		II	0.07	0.04	0.03	6.2
		III	0.10	0.04	0.06	10.3
	Frozen	I	0.09	0.04	0.05	8.6
		II	0.11	0.04	0.07	12.1
		III	0.04		0.04	6.9
	Canned	I	0.14	0.01	0.13	22.4
		II	0.14	0.02	0.12	20.7
		III	0.09	0.03	0.06	10.3
	Avg. 13.4					
Carrots (West Coast)	Unwashed	I	0.06	0.03	0.03	6.2
		II	0.11	0.04	0.07	7.0
		III	0.10	0.02	0.08	13.8
	Washed	I	0.12	0.03	0.09	16.6
		II	0.03	--	0.03	6.2
		III	0.12	0.04	0.08	13.8
	Diced	I	0.11	0.04	0.07	12.1
		II	0.11	0.02	0.09	16.6
		III	0.12	0.02	0.10	17.2
	Frozen	I	0.12	0.05	0.07	12.1
		II	0.12	0.05	0.07	12.1
		III	0.13	0.04	0.11	19.0
	Canned	I	0.12	0.04	0.08	13.8
		II	0.16	0.05	0.11	19.0
		III	0.13	0.04	0.09	16.6
	Avg. 13.1					

Table IV. (Continued)

Commodity (Location)	Process Stage	Replicate	ETU Found	ETU in check + 0.05 ETU	ETU from EBDC	% Conversion of EBDC to ETU
Spinach (East Coast)	Unwashed	I	0.14	0.03	0.11	19.0
		II	0.10	0.03	0.07	12.1
		III	0.11	0.04	0.07	12.1
	Washed	I	0.11	0.04	0.07	12.1
		II	0.06	0.04	0.02	3.4
		III	0.21	0.07	0.14	24.1
	Frozen	I	0.11	0.03	0.08	13.8
		II	0.13	0.04	0.09	15.5
		III	0.13	0.04	0.09	15.5
	Canned	I	0.16	0.04	0.12	20.7
		II	0.20	0.04	0.16	27.6
		III	0.16	0.03	0.13	22.4
	Avg. 16.5					
Spinach (West Coast)	Unwashed	I	0.04	0.03	0.01	2.0
		II	0.06	0.01	0.05	10.2
		III	0.04	0.03	0.01	2.0
	Washed	I	0.06	0.04	0.02	4.0
		II	0.04	0.03	0.01	2.0
		III	0.04	0.03	0.01	2.0
	Frozen	I	0.07	0.01	0.06	12.2
		II	0.07	0.04	0.03	6.1
		III	0.07	0.03	0.04	8.2
	Canned	I	0.13	0.05	0.08	16.3
		II	0.08	0.05	0.03	6.1
		III	0.14	0.06	0.08	16.3
	Avg. 7.3					

¹ 1.2 ppm EBDC (except 1.0 ppm for West Coast spinach) and 0.05 ppm ETU were added to each sample. Theoretical ETU added is $1.2 \times 0.486 + 0.05 = 0.63$ ppm; ($1.0 \times 0.486 + 0.05 = 0.54$ ppm for West Coast spinach). ETU in checks, fortified only with 0.05 ppm ETU, were subtracted from ETU found to give ETU from EBDC. Percent conversion was calculated by dividing values in the "ETU from EBDC" column by 0.58 (or 0.49 for West Coast spinach) $\times 100$.

Table V. Conversion of EBDC¹ (Added as Dithane M-22[®])
to ETU on Control Samples of Tomatoes and
Spinach--at Various Processing Stages

Commodity (Location)	Process Stage	Replicate	ETU Found	ETU in check + 0.05 ETU	ETU from EBDC	% Conversion of EBDC to ETU
Tomatoes (West Coast)	Unwashed	I	0.08	0.04	0.04	7.4
		II	0.09	0.06	0.03	6.6
		III	0.05	0.04	0.01	1.7
	Washed	I	0.20	0.06	0.14	25.9
		II	0.13	0.04	0.09	16.7
		III	0.31	0.04	0.27	50.0
	Canned Whole	I	0.16	0.04	0.12	22.2
		II	0.17	0.06	0.11	20.3
		III	0.13	0.04	0.09	16.7
	Juice	I	0.19	0.06	0.14	25.9
		II	0.20	0.06	0.15	27.8
		III	0.15	0.06	0.10	18.5
	Avg. 19.9					
Spinach (West Coast)	Unwashed	I	0.08	0.04	0.04	7.4
		II	0.03	0.03	----	---
		III	0.06	0.02	0.04	7.4
		I	0.06	0.03	0.03	6.6
		II	0.06	0.02	0.04	6.9
		III	0.08	0.03	0.06	9.3
		I	0.04	0.03	0.01	1.9
		II	0.05	0.03	0.02	3.7
		III	0.06	0.02	0.03	6.6
		I	0.07	0.03	0.04	7.4
		II	0.04	0.03	0.01	1.9
		III	0.07	0.03	0.04	7.4
	Avg. 5.4					

¹ 1.2 ppm EBDC and 0.05 ppm ETU were added to each sample. Theoretical ETU added is $1.1 \times 0.486 + 0.05 = 0.59$ ppm. ETU in checks, fortified only with 0.05 ppm ETU, were subtracted from ETU found to give ETU from EBDC. Percent conversion was calculated by dividing values in the "ETU from EBDC" column by 0.54×100 .

Table VI. Rate of Formation and Breakdown of ETU from
Technical EBDC (6.2 ppm ai) in Boiling
Distilled-Water Buffered at pH 5.21

Sample	Amt. Found EBDC (ppm)	Corrected for % Recovery	Avg.	Amt. Found ETU (ppm)	Corrected for % Recovery	Avg.	% Conversion of EBDC to ETU
0.5 min.	2.6, 2.6	3.8, 3.9	3.8	1.01, 1.39	1.01, 1.39	1.20	107
30. min.	0.2, 0.2	0.3, 0.3	0.3	1.78, 1.76	1.78, 1.76	1.77	61
60. min.	0.1, 0.2	0.2, 0.3	0.3	1.72, 0.63	1.72, 0.63	1.72	59
90. min.	<0.1, <0.1	<0.1, <0.1	<0.1	2.61, 2.92	2.61, 2.92	2.72	90

Recovery Studies EBDC (ppm)

Sample	Amount Added	Amount Found	Corrected for Check*	% Recovery
0 min. control	none	<0.1	--	--
"	none	<0.1	--	--
"	0.6**	0.4	0.4	67
"	0.6**	0.4	0.4	67
60 min. control	none	<0.1	--	--
"	none	<0.1	--	--
"	0.6**	0.4	0.4	67
"	0.6**	0.4	0.4	67

Recovery Studies ETU (ppm)

Sample	Amount Added	Amount Found	Corrected for Check*	% Recovery
0 min. control	none	<0.01	--	--
"	0.05	0.05	0.05	100
60 min. control	none	<0.01	--	--
"	0.05	0.05	0.05	100

*Corrected by subtraction of amount found in check sample.

**Amount added has been corrected for % activity of 62%.
Determination done 1 week following other determinations.
Value excluded from subsequent calculations.

Calculation of conversion of EBDC to ETU:

Amt. EBDC fortified - amt. EBDC found $\times$ 0.486 = A

Amt. ETU found - amt. ETU (check) = B

B/A = Conversion EBDC to ETU

Example:

0.5 min. A = (6.2 - 2.6) 0.486 = 1.12

B = 1.20

B/A = $\frac{1.20}{1.12} = 1.07 \times 100 = 107\%$

Table VII-1-A. Residues of EBDC (from M-22) in West Coast
Washed Tomatoes Fortified with 0.5 ppm EBDC¹

Sample	Amount Found EBDC (ppm)	*Corrected for Check	Corrected for Recovery
Check	<0.1, <0.1, <0.1, <0.1, <0.1, <0.1		
1	0.2	0.2	0.2
2	0.2	0.2	0.2
3	0.2	0.2	0.2
4	0.1	0.1	0.1
5	0.2	0.2	0.2
6	0.2	0.2	0.2

Laboratory Recovery Studies EBDC (ppm)

	Amount Added	Amount Found	*Corrected for Check	% Recovery
Check	1.2 EBDC ²	1.0	1.0	83

Average recovery of fortification with 0.5 ppm added EBDC to washed tomatoes was 80%.

*Corrected by subtraction of amount found in check sample.

¹Fortified with 0.5 ppm EBDC after washing in the pilot plant. Amount added has been corrected for 60% activity.

²Fortified with 1.2 ppm EBDC in the laboratory. Amount added has been corrected for 60% activity.

Table VII-1-B. Residues of EBDC (from M-22) in West Coast
Canned Whole Tomatoes Fortified with 0.5 ppm EBDC¹

<u>Sample</u>	<u>Amount Found EBDC (ppm)</u>	<u>*Corrected for Check</u>	<u>Corrected for Recovery</u>
Check	<0.1, <0.1, <0.1, <0.1, <0.1		
1	0.1	0.1	0.1
2	0.1	0.1	0.1
3	0.1	0.1	0.1
4	0.1	0.1	0.1
5	0.1	0.1	0.1
6	0.1	0.1	0.1

Laboratory Recovery Studies EBDC (ppm)

	<u>Amount Added</u>	<u>Amount Found</u>	<u>*Corrected for Check</u>	<u>% Recovery</u>
Check	1.2 EBDC ²	0.9	0.9	75

Average recovery of EBDC in canned tomatoes which were prepared from washed tomatoes containing added EBDC at 0.5 ppm was 20%.

*Corrected by subtraction of amount found in check sample.

¹Fortified with 0.5 ppm EBDC after washing in the pilot plant. Amount added has been corrected for 60% activity.

²Fortified with 1.2 ppm EBDC in the laboratory. Amount added has been corrected for 60% activity.

Table VII-1-C. Residues of EBDC (from M-22) in West Coast
Tomato Juice Fortified with 0.5 ppm EBDC¹

<u>Sample</u>	<u>Amount Found EBDC (ppm)</u>	<u>*Corrected for Check</u>	<u>Corrected for Recovery</u>
Check	<0.1, <0.1, <0.1, <0.1, <0.1, <0.1		
1	0.3	0.3	0.4
2	0.3	0.3	0.4
3	0.3	0.3	0.4
4	0.3	0.3	0.4
5	0.3	0.3	0.4
6	0.3	0.3	0.4

Laboratory Recovery Studies EBDC (ppm)

	<u>Amount Added</u>	<u>Amount Found</u>	<u>*Corrected for Check</u>	<u>% Recovery</u>
Check	1.2 EBDC ²	0.9	0.9	75

The average % recovery of EBDC in canned tomato juice prepared from washed tomatoes containing added EBDC at 0.5 ppm was 80%.

*Corrected by subtraction of amount found in check sample.

¹Fortified with 0.5 ppm EBDC after washing in the pilot plant. Amount added has been corrected for 60% activity.

²Fortified with 1.2 ppm EBDC in the laboratory. Amount added has been corrected for 60% activity.

Table VII-1-D. Residues of EBDC (from M-22) in West Coast
Tomato Sauce Fortified with 0.5 ppm EBDC¹

<u>Sample</u>	<u>Amount Found EBDC (ppm)</u>		<u>*Corrected for Check</u>	<u>Corrected for Recovery</u>
Check	0.3	0.3		
1		1.5	1.2	1.3
2		1.5	1.2	1.3
3		1.9	1.6	1.7
4		1.7	1.4	1.5
5		1.8	1.5	1.6
6		1.5	1.2	1.3

Laboratory Recovery Studies EBDC (ppm)

	<u>Amount Added</u>	<u>Amount Found</u>	<u>*Corrected for Check</u>	<u>% Recovery</u>
Check	1.2 EBDC ²	1.4	1.1	92
Check	1.2 EBDC	1.4	1.1	92

*Corrected by subtraction of amount found in check sample.

¹Fortified with 0.5 ppm EBDC after washing in the pilot plant.
Amount added has been corrected for 60% activity.

²Fortified with 1.2 ppm EBDC in the laboratory. Amount added
has been corrected for 60% activity.

Table VII-1-E. Residues of EBDC (from M-22) in West Coast
Tomato Paste Fortified with 0.5 ppm EBDC¹

<u>Sample</u>	<u>Amount Found EBDC (ppm)</u>		<u>*Corrected for Check</u>	<u>Corrected for Recovery</u>
Check	<0.1	<0.1		
1	2.7		2.7	2.4
2	2.7		2.7	2.4
3	2.7		2.7	2.4
4	2.9		2.9	2.6
5	2.8		2.8	2.5
6	2.8		2.8	2.5

Laboratory Recovery Studies EBDC (ppm)

	<u>Amount Added</u>	<u>Amount Found</u>	<u>*Corrected for Check</u>	<u>% Recovery</u>
Check	1.2 EBDC ²	1.3	1.3	108
Check	1.2 EBDC	1.4	1.4	117

*Corrected by subtraction of amount found in check sample.

¹Fortified with 0.5 ppm EBDC after washing in the pilot plant.
Amount added has been corrected for 60% activity.

²Fortified with 1.2 ppm EBDC in the laboratory. Amount added
has been corrected for 60% activity.

Table VII-2-A. Residues of ETU (from M-22) in West Coast
Washed Tomatoes Fortified with 0.5 ppm EBDC¹

<u>Sample</u>	<u>Amount Found ETU (ppm)</u>	<u>*Corrected for Check</u>	<u>Corrected for Recovery</u>
Check	0.02, 0.02, 0.01, 0.03, 0.03, 0.03		
1	0.09	0.07	0.09
2	<0.01	<0.01	<0.01
3	0.02	<0.01	<0.01
4	0.04	0.02	0.03
5	0.02	<0.01	<0.01
6	0.02	<0.01	<0.01

Laboratory Recovery Studies ETU (ppm)

	<u>Amount Added</u>	<u>Amount Found</u>	<u>*Corrected for Check</u>	<u>% Recovery</u>
Check	0.05 ETU ²	0.06	0.04	80

*Corrected by subtraction of amount found in check sample.

¹Fortified with 0.5 ppm EBDC after washing in the pilot plant.
Amount added has been corrected for 60% activity.

²Fortified with 0.05 ppm ETU in the Laboratory.

Table VII-2-B. Residues of ETU (from M-22) in West Coast
Canned Whole Tomatoes Fortified with 0.5 ppm EBDC¹

<u>Sample</u>	<u>Amount Found ETU (ppm)</u>	<u>*Corrected for Check</u>	<u>Corrected for Recovery</u>
Check	0.03, 0.02, 0.03, 0.03, 0.03, 0.02		
1	0.06	0.02	0.03
2	0.04	<0.01	<0.01
3	0.06	0.03	0.06
4	0.06	0.02	0.03
5	0.06	0.02	0.03
6	0.06	0.02	0.03

Laboratory Recovery Studies ETU (ppm)

	<u>Amount Added</u>	<u>Amount Found</u>	<u>*Corrected for Check</u>	<u>% Recovery</u>
Check	0.05 ETU ²	0.06	0.03	60

*Corrected by subtraction of amount found in check sample.

¹ Fortified with 0.5 ppm EBDC after washing in the pilot plant.
Amount added has been corrected for 60% activity.

² Fortified with 0.05 ppm ETU in the Laboratory.

Table VII-2-C. Residues of ETU (from M-22) in West Coast
Tomato Juice Fortified with 0.5 ppm EBDC¹

<u>Sample</u>	<u>Amount Found ETU (ppm)</u>	<u>*Corrected for Check</u>	<u>Corrected for Recovery</u>
Check	<0.01, 0.03, 0.03, 0.02, 0.04		
1	0.09	0.07	0.09
2	0.09	0.07	0.09
3	0.07	0.06	0.06
4	0.10	0.08	0.10
5	0.09	0.07	0.09
6	0.09	0.07	0.09

Laboratory Recovery Studies ETU (ppm)

	<u>Amount Added</u>	<u>Amount Found</u>	<u>*Corrected for Check</u>	<u>% Recovery</u>
Check	0.05 ETU ²	0.06	0.04	80

*Corrected by subtraction of amount found in check sample.

¹Fortified with 0.5 ppm EBDC after washing in the pilot plant.
Amount added has been corrected for 60% activity.

²Fortified with 0.05 ppm ETU in the Laboratory.

Table VII-2-D. Residues of ETU (from M-22) in West Coast
Tomato Sauce Fortified with 0.5 ppm EBDC¹

Sample	Amount Found ETU (ppm)	*Corrected for Check	Corrected for Recovery
Check	0.03, 0.02, 0.01, 0.03, 0.03, 0.03		
1	0.07	0.04	0.05
2	0.09	0.06	0.07
3	0.09	0.06	0.07
4	0.07	0.04	0.05
5	0.09	0.06	0.07
6	0.08	0.05	0.06

Laboratory Recovery Studies ETU (ppm)

	Amount Added	Amount Found	*Corrected for Check	% Recovery	Avg.
Check	0.05 ETU ²	0.07	0.04	80	87
Check	0.05 ETU	0.07	0.04	80	
Check	0.05 ETU	0.08	0.05	100	

*Corrected by subtraction of amount found in check sample.

¹Fortified with 0.5 ppm EBDC after washing in the pilot plant.
Amount added has been corrected for 60% activity.

²Fortified with 0.05 ppm ETU in the Laboratory.

Table VII-2-E. Residues of ETU (from M-22) in West Coast
Tomato Paste Fortified with 0.5 ppm EBDC¹

Sample	Amount Found ETU (ppm)	Avg.	*Corrected for Check	Corrected for Recovery
Check	0.01, 0.04, 0.40	0.03		
1	0.20		0.17	0.26
2	0.03		<0.01	<0.01
3	0.16		0.16	0.22
4	0.13		0.10	0.16
5	0.13		0.10	0.16
6	0.19		0.16	0.24

Laboratory Recovery Studies ETU (ppm)

	Amount Added	Amount Found	*Corrected for Check	% Recovery	Avg.
Check	0.10 ETU ²	0.11	0.08	80	67
Check	0.05 ETU	0.06	0.03	60	
Check	0.05 ETU	0.06	0.03	60	

*Corrected by subtraction of amount found in check sample.

¹Fortified with 0.5 ppm EBDC after washing in the pilot plant.
Amount added has been corrected for 60% activity.

²Fortified with 0.05 ppm ETU in the Laboratory.

Table VII-3-A. Residues of ETU (from M-22) in West Coast
Washed Tomatoes Fortified with 0.07 ppm ETU¹

<u>Sample</u>	<u>Amount Found ETU (ppm)</u>	<u>*Corrected for Check</u>	<u>Corrected for Recovery</u>
Check			
1	0.03	0.01	0.01
2	0.02	0.01	0.01
3	0.01	0.01	0.01
4	0.02	0.01	0.01
5	0.03	0.01	0.01
6	0.03	0.01	0.01

See Table VII-2-A for Recovery Studies.

*Corrected by subtraction of amount found in check sample.

¹Fortified with 0.07 ppm ETU after washing in the pilot plants.

Table VII-3-B. Residues of ETU (from M-22) in West Coast
Canned Whole Tomatoes Fortified with 0.07 ppm ETU¹

<u>Sample</u>	<u>Amount Found ETU (ppm)</u>	<u>*Corrected for Check</u>	<u>Corrected for Recovery</u>
Check			
1	0.03	<0.01	<0.01
2	0.05	0.02	0.03
3	0.02	<0.01	<0.01
4	0.02	<0.01	<0.01
5	0.02	<0.01	<0.01
6	0.02	<0.01	<0.01

See Table VII-2-B for Recovery Studies.

*Corrected by subtraction of amount found in check sample.

¹Fortified with 0.07 ppm ETU after washing in the pilot plants.

Table VII-3-C. Residues of ETU (from M-22) in West Coast
Tomato Juice Fortified with 0.07 ppm ETU¹

<u>Sample</u>	<u>Amount Found ETU (ppm)</u>	<u>*Corrected for Check</u>	<u>Corrected for Recovery</u>
Check			
1	0.05	0.03	0.04
2	0.03	0.01	0.01
3	0.09	0.07	0.09
4	0.08	0.06	0.08
5	0.06	0.04	0.05
6	0.04	0.02	0.03

See Table VII-2-C for Recovery Studies.

*Corrected by subtraction of amount found in check sample.

¹Fortified with 0.07 ppm ETU after washing in the pilot plants.

Table VII-3-D. Residues of ETU (from M-22) in West Coast
Tomato Sauce Fortified with 0.07 ppm ETU¹

<u>Sample</u>	<u>Amount Found ETU (ppm)</u>	<u>*Corrected for Check</u>	<u>Corrected for Recovery</u>
Check			
1	0.17	0.14	0.18
2	0.19	0.16	0.20
3	0.14	0.11	0.14
4	0.19	0.16	0.20
5	0.18	0.15	0.19
6	0.18	0.15	0.19

See Table VII-2-D for Recovery Studies.

*Corrected by subtraction of amount found in check sample.

¹Fortified with 0.07 ppm ETU after washing in the pilot plants.

Table VII-3-E. Residues of ETU (from M-22) in West Coast
Tomato Paste Fortified with 0.07 ppm ETU¹

<u>Sample</u>	<u>Amount Found ETU (ppm)</u>	<u>*Corrected for Check</u>	<u>Corrected for Recovery</u>
Check			
1	0.16	0.13	0.19
2	0.18	0.15	0.22
3	0.13	0.10	0.16
4	0.21	0.18	0.27
5	0.18	0.15	0.22
6	0.26	0.22	0.33

See Table VII-2-E for Recovery Studies.

*Corrected by subtraction of amount found in check sample.

¹Fortified with 0.07 ppm ETU after washing in the pilot plants.

Table VII-4-A. Residues of EBDC (From M-22) in West Coast washed
Tomatoes fortified with 0.5 ppm EBDC + 0.07 ppm ETU¹

<u>Sample</u>	<u>Amount Found EBDC (ppm)</u>	<u>*Corrected for Check</u>	<u>Corrected for Recovery</u>
Check			
1	0.1	0.1	0.1
2	0.2	0.2	0.3
3	0.3	0.3	0.4
4	0.2	0.2	0.3
5	0.2	0.2	0.3
6	0.2	0.2	0.3

Laboratory Recovery Studies EBDC (ppm)

	<u>Amount Added</u>	<u>Amount Found</u>	<u>*Corrected for Check</u>	<u>% Recovery</u>
Check	1.2 EBDC ²	0.9	0.9	75

The average % recovery of added EBDC to washed tomatoes was 60%.

*Corrected by subtraction of amount found in check sample.

¹Fortified with 0.5 ppm EBDC + 0.07 ppm ETU after washing in the pilot plant. EBDC added has been corrected for 60% activity.

²Fortified with 1.2 ppm EBDC in the laboratory. Amount added has been corrected for 60% activity.

Table VII-4-B. Residues of EBDC (from M-22) in West Coast Canned Whole Tomatoes Fortified with 0.5 ppm EBDC + 0.07 ppm ETU¹

<u>Sample</u>	<u>Amount Found EBDC (ppm)</u>	<u>*Corrected for Check</u>	<u>Corrected for Recovery</u>
Check			
1	0.1	0.1	0.1
2	0.1	0.1	0.1
3	0.1	0.1	0.1
4	0.1	0.1	0.1
5	0.1	0.1	0.1
6	0.1	0.1	0.1

Laboratory Recovery Studies EBDC (ppm)

	<u>Amount Added</u>	<u>Amount Found</u>	<u>*Corrected for Check</u>	<u>% Recovery</u>
Check	1.2 EBDC ²	1.2	1.2	100

The average % recovery of added EBDC from canned whole tomatoes prepared from fortified washed tomatoes was 20%. The preparation step calls for removal of skins.

*Corrected by subtraction of amount found in check sample.

¹ Fortified with 0.5 ppm EBDC + 0.07 ppm ETU after washing in the pilot plant. EBDC added has been corrected for 60% activity.

² Fortified with 1.2 ppm EBDC in the laboratory. Amount added has been corrected for 60% activity.

Table VII-4-C. Residues of EBDC (from M-22) in West Coast Canned
Tomato Juice Fortified with 0.5 ppm EBDC + 0.07 ppm ETU¹

Sample	Amount Found EBDC (ppm)	*Corrected for Check	Corrected for Recovery
Check			
1	0.4	0.4	0.4
2	0.3	0.3	0.3
3	0.4	0.4	0.4
4	0.4	0.4	0.4
5	0.4	0.4	0.4
6	0.4	0.4	0.4

Laboratory Recovery Studies EBDC (ppm)

	Amount Added	Amount Found	*Corrected for Check	% Recovery
Check	1.2 EBDC ²	1.3	1.3	100

The average recovery of added EBDC from tomato juice prepared from
fortified washed tomatoes was 80%.

*Corrected by subtraction of amount found in check sample.

¹ Fortified with 0.5 ppm EBDC + 0.07 ppm ETU after washing in the
pilot plant. EBDC added has been corrected for 60% activity.

² Fortified with 1.2 ppm EBDC in the laboratory. Amount added has
been corrected for 60% activity.

Table VII-4-D. Residues of EBDC (from 4-22) in West Coast Tomato
Sauce Fortified with 0.5 ppm EBDC + 0.07 ppm ETU¹

<u>Sample</u>	<u>Amount Found EBDC (ppm)</u>	<u>*Corrected for Check</u>	<u>Corrected for Recovery</u>
Check			
1	1.9	1.6	1.7
2	1.9	1.6	1.7
3	1.9	1.6	1.7
4	1.9	1.6	1.7
5	1.8	1.5	1.6
6	1.9	1.6	1.7

See Table VII-2-D for Recovery Studies.

*Corrected by subtraction of amount found in check sample.

¹Fortified with 0.5 ppm EBDC + 0.07 ppm ETU after washing in the pilot plant. EBDC added has been corrected for 60% activity.

Table VII-4-E. Residues of EBDC (from M-22) in West Coast Tomato
Paste Fortified with 0.5 ppm EBDC + 0.07 ppm ETU¹

<u>Sample</u>	<u>Amount Found EBDC (ppm)</u>	<u>*Corrected for Check</u>	<u>Corrected for Recovery</u>
Check			
1	3.1	3.1	3.7
2	4.0	4.0	4.2
3	4.0	4.0	4.2
4	4.2	4.2	3.7
5	4.4	4.4	3.9
6	4.5	4.5	4.0

See Table VII-2-E for Recovery Studies.

*Corrected by subtraction of amount found in check sample.

¹Fortified with 0.5 ppm EBDC + 0.07 ppm ETU after washing in the
pilot plant. EBDC added has been corrected for 60% activity.

Table VII-5-A. Residues of ETU (from M-22) in West Coast Washed
Tomatoes Fortified with 0.5 ppm EBDC + 0.07 ppm ETU¹

<u>Sample</u>	<u>Amount Found ETU (ppm)</u>	<u>*Corrected for Check</u>	<u>Corrected for Recovery</u>
Check			
1	0.06	0.03	0.04
2	0.01	<0.01	<0.01
3	0.02	<0.01	<0.01
4	0.03	0.01	0.01
5	0.03	0.01	0.01
6	0.02	<0.01	<0.01

See Table VII-2-A for Recovery Studies.

*Corrected by subtraction of amount found in check sample.

¹Fortified with 0.5 ppm EBDC + 0.07 ppm ETU after washing in the pilot plant. EBDC added has been corrected for 60% activity.

Table VII-5-B. Residues of ETU (from M-22) in West Coast Canned
Whole Tomatoes Fortified with 0.5 ppm EBDC + 0.07 ppm ETU¹

<u>Sample</u>	<u>Amount Found ETU (ppm)</u>	<u>*Corrected for Check</u>	<u>Corrected for Recovery</u>
Check			
1	0.06	0.03	0.05
2	0.07	0.04	0.07
3	0.07	0.04	0.07
4	0.07	0.04	0.07
5	0.05	0.02	0.03
6	0.08	0.05	0.06

See Table VII-2-B for Recovery Studies.

*Corrected by subtraction of amount found in check sample.

¹ Fortified with 0.5 ppm EBDC + 0.07 ppm ETU after washing in
the pilot plant. EBDC added has been corrected for 60%
activity.

Table VII-5-C. Residues of ETU (from M-22) in West Coast Tomato
Juice Fortified with 0.5 ppm EBDC + 0.07 ppm ETU¹

<u>Sample</u>	<u>Amount Found ETU (ppm)</u>	<u>*Corrected for Check</u>	<u>Corrected for Recovery</u>
Check			
1	0.08	0.06	0.08
2	0.14	0.12	0.15
3	0.13	0.11	0.14
4	0.12	0.10	0.13
5	0.06	0.04	0.05
6	0.07	0.05	0.06

See Table VII-2-C for Recovery Studies.

*Corrected by subtraction / amount found in check sample.

¹Fortified with 0.5 ppm EBDC + 0.07 ppm ETU after washing in the pilot plant. EBDC added has been corrected for 60% activity.

Table VII-5-D. Residues of ETU (from M-22) in West Coast Tomato
Sauce Fortified with 0.5 ppm EBDC + 0.07 ppm ETU¹

<u>Sample</u>	<u>Amount Found ETU (ppm)</u>	<u>*Corrected for Check</u>	<u>Corrected for Recovery</u>
Check			
1	0.40	0.37	0.46
2	0.26	0.23	0.29
3	0.29	0.26	0.33
4	0.36	0.33	0.41
5	0.24	0.21	0.26
6	0.56	0.53	0.66

See Table VII-2-D for Recovery Studies.

*Corrected by subtraction of amount found in check sample.

¹Fortified with 0.5 ppm EBDC + 0.07 ppm ETU after washing in the pilot plant. EBDC added has been corrected for 60% activity.

Table VII-5-E. Residues of ETU (from M-22) in West Coast Tomato
Paste Fortified with 0.5 ppm EBDC + 0.07 ppm ETU¹

<u>Sample</u>	<u>Amount Found ETU (ppm)</u>	<u>*Corrected for Check</u>	<u>Corrected for Recovery</u>
Check			
1	0.62	0.59	0.88
2	0.19	0.16	0.24
3	0.63	0.60	0.90
4	0.41	0.38	0.67
5	0.51	0.48	0.72
6	0.22	0.19	0.28

See Table VII-2-E for Recovery Studies.

*Corrected by subtraction of amount found in check sample.

¹Fortified with 0.5 ppm EBDC + 0.07 ppm ETU after washing in the pilot plant. EBDC added has been corrected for 60% activity.

Table VII. Summary Table of Average Residues (ppm) from Fortified Control Whole Tomatoes and from Whole Tomatoes with Residues of the Same Magnitude from Field-Applied Dithane M-22[®] 4x Treatment--at Various Stages of Processing

Process Stage	Fort. with 0.5 ppm EBDC		Fort. with 0.07 ppm ETU	Fort. with 0.5 ppm EBDC and 0.07 ppm ETU		4x treatment in field	
	EBDC	ETU		EBDC	ETU	EBDC	ETU
Washed Tomatoes	0.2	0.02	0.01	0.3	0.02	0.5	0.10
Canned Whole Tomatoes	0.1	0.03	0.01	0.1	0.06	0.4	0.07
Canned Tomato Juice	0.4	0.09	0.08	0.4	0.10	0.8	0.12
Tomato Sauce	1.5	0.06	0.18	1.8	0.40	2.7	0.22
Tomato Paste	2.5	0.17	0.23	3.8	0.60	3.7	0.46

Results for EBDC have been corrected for β activity; all results have been corrected for check values and recovery data.

Table VIII. Summary of all Recovery Data

(Dithane M-45[®] and Dithane M-22[®])

Recoveries From Dithane M-45[®]

<u>East Coast</u>			<u>West Coast</u>		
<u>Tomatoes</u>	<u>EBDC</u>	<u>ETU</u>	<u>Tomatoes</u>	<u>EBDC</u>	<u>ETU</u>
Unwashed	108	87	Unwashed	96	87
Washed	97	80	Washed	89	60
Tailings	95	47	Tailings	--	37
Canned	90	107	Canned	68	73
(Average)	95.2	80	(Average)	84.2	64.2
<u>Carrots</u>	<u>EBDC</u>	<u>ETU</u>	<u>Carrots</u>	<u>EBDC</u>	<u>ETU</u>
Unwashed	82	80	Unwashed	82	60
Washed	71	73	Washed	79	67
Diced	87	73	Diced	81	53
Frozen	82	80	Frozen	88	93
Canned	80	40	Canned	83	87
(Average)	80.9	69.3	(Average)	82.6	72
<u>Spinach</u>	<u>EBDC</u>	<u>ETU</u>	<u>Spinach</u>	<u>EBDC</u>	<u>ETU</u>
Unwashed	68	73	Unwashed	87	47
Washed	85	80	Washed	67	67
Frozen	86	73	Frozen	79	53
Canned	90	67	Canned	76	107
(Average)	86.7	70.9	(Average)	77.3	68.3

Average % Recovery East Coast All Commodities

<u>EBDC</u>	<u>ETU</u>
87.9	73.2

Average % Recovery West Coast All Commodities

<u>EBDC</u>	<u>ETU</u>
81.2	68.9

Average % Recovery East and West Coast All Commodities

<u>EBDC</u>	<u>ETU</u>
n=211 85.6	n=77 70.8

Average % Recovery by Commodity

	<u>EBDC</u>	<u>ETU</u>
Tomatoes	91.8	72.0
Carrots	81.0	70.7
Spinach	84.3	69.6

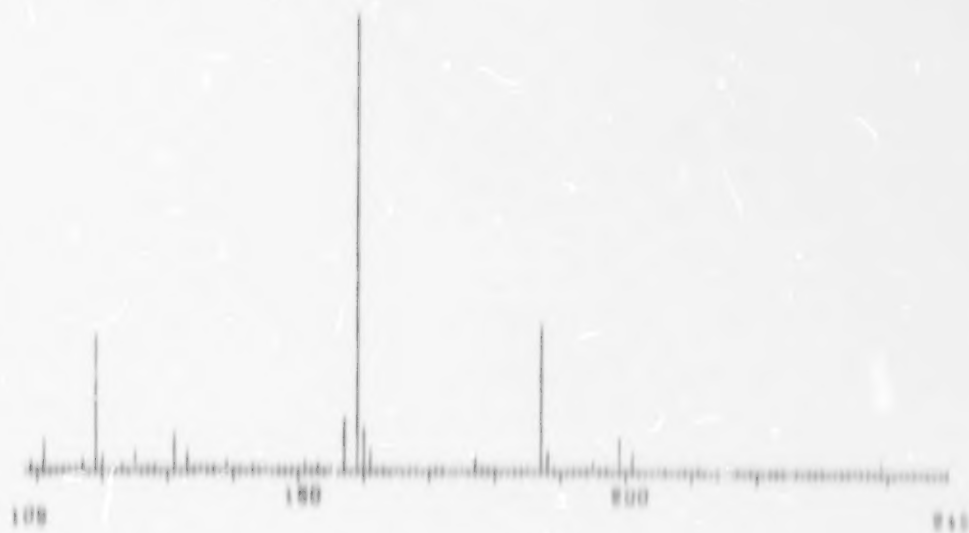
Recoveries From Dithane M-22[®] (West Coast only)

	<u>EBDC</u>	<u>ETU</u>
Spinach	72.6	47.6
Tomatoes	72.2	63.6

APPENDIX E

GC/MS Confirmatory Data

APPENDIX E TABLE I
MASS SPECTRA OF STANDARD ETU
SAMPLE: S-NBUTYL DERIVATIVE OF ETU
CI MODE



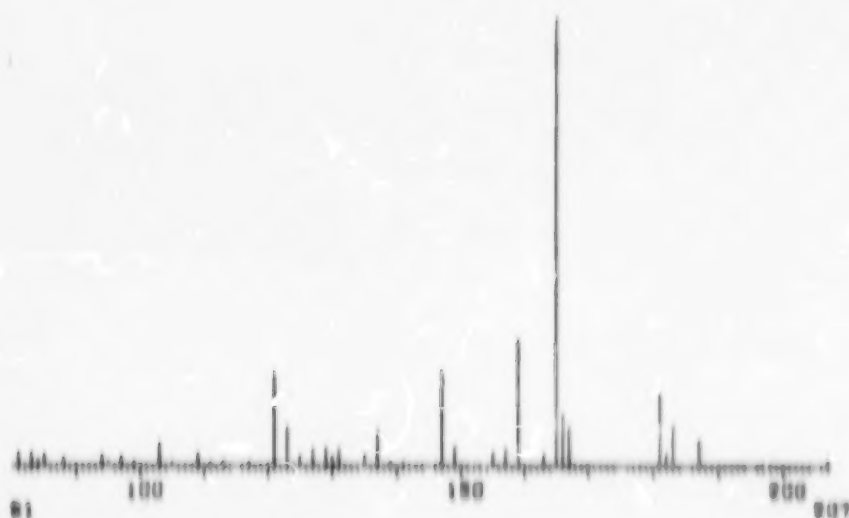
FRN 8799 SPECT 22

APPENDIX E TABLE II

MASS SPECTRA OF CANNED SPINACH

SAMPLE: CONTROL REP III FOR S-N BUTYL ETU

CI MODE (CH_4)



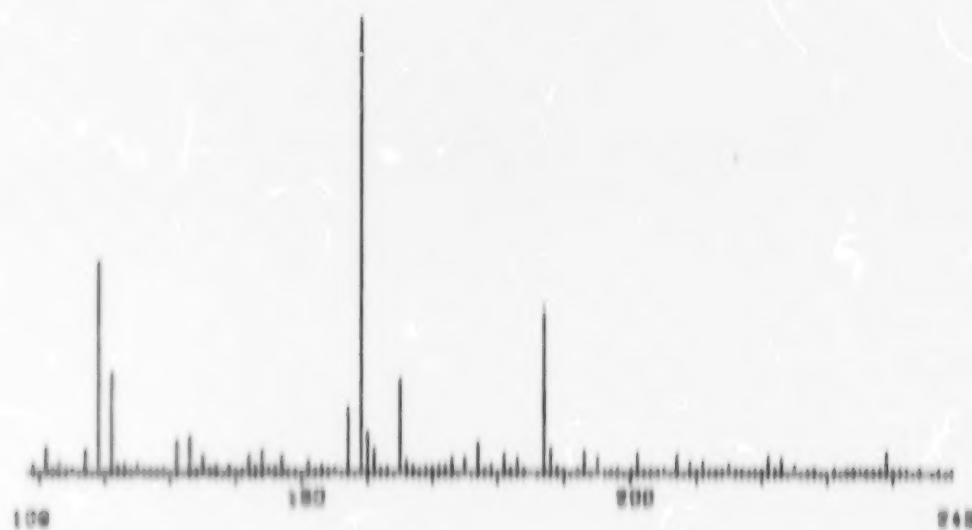
FRN 8404 SPEC1 1

APPENDIX E TABLE III

MASS SPECTRA OF CANNED SPINACH M-45

SAMPLE: 1X REP I FOR 5-NBUTYL ETU

CI MODE (CH₄)



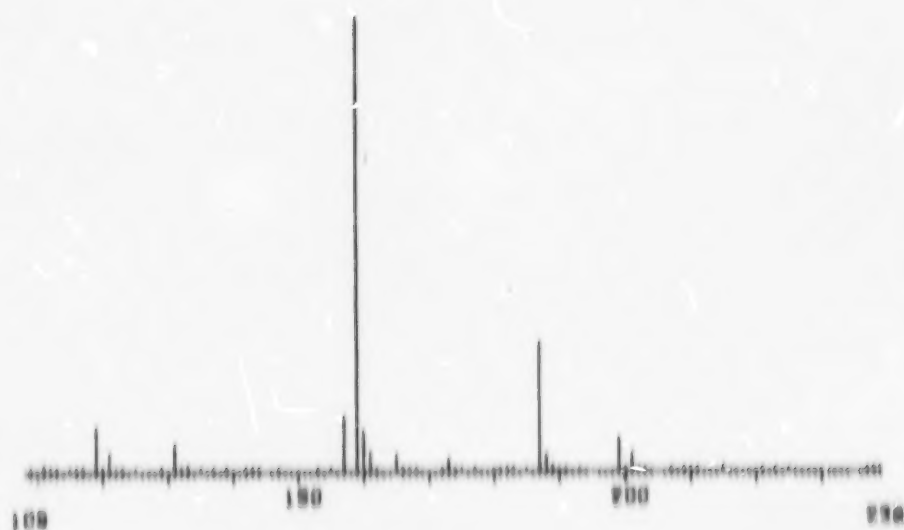
FRN 8801 SPECT 21

APPENDIX E TABLE IV

MASS SPECTRA FOR CANNED SPINACH M-45

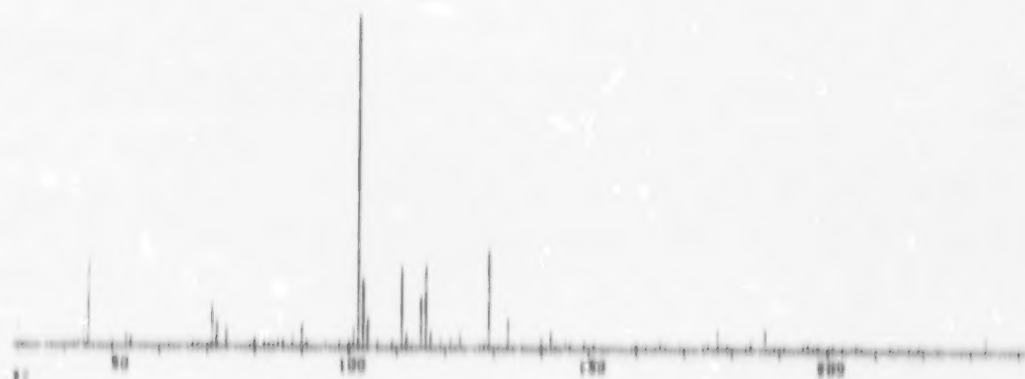
SAMPLE: 4X REP III FOR S-NBUTYL ETU

CI MODE. (CH₄)



FRN 8800 SPECT 21

APPENDIX E TABLE V
MASS SPECTRA OF STANDARD ETU
SAMPLE: 5-NBUTYL DERIVATIVE OF ETU
EI MODE

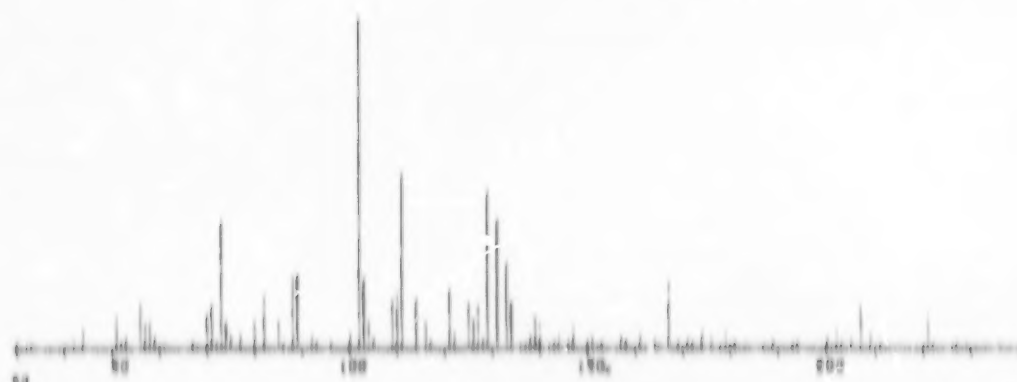


KRN 8557 SPECT 1

APPENDIX E TABLE VI
 MASS SPECTRA OF CHARGED SPRAYED COPIES
 SAMPLE: CONTROL COPY III FROM 1-400TH, 2TH
 121-4002

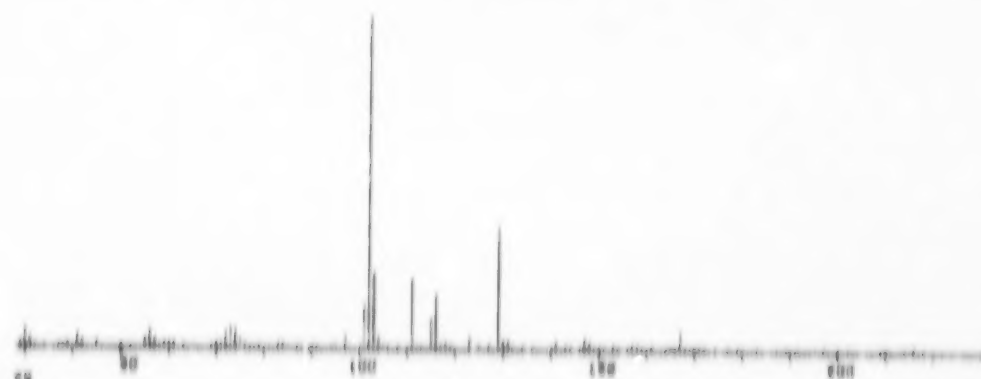


APPENDIX 2 TABLE VII
MASS SPECTRA OF CANNED SPINACH M-48
SAMPLE: 1X REP 1 FOR S-NBUTYL ETU
EI MODE



PMN 8555 SPECT 1

APPENDIX E TABLE VIII
GAS SPECTRA OF CANNED SPINACH R-45
SAMPLE: 4X REP III FOR S-NBUTYL ETU
EI MODE



FRN 0504 SPECT 1